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WADC TECHNICAL REPORT 55-18
PART I

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DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS-CONCENTRATIONS AND HYDROGEN EMBRITTLEMENT

Part I. Effects of Hydrogen Embrittlement on Static Properties of High-Strength Steels

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MATERIALS LABORATORY
CONTRACT No. AF 33(616)-2362
PROJECT No. 7360

WRIGHT AIR DEVELOPMENT CENTER
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

Carpenter Litho & Prtg. Co., Springfield, O.
300 - 22 November 1955

FOREWORD

This report was prepared by Syracuse University, under USAF Contract No. AF 33(616)-2362. The contract was initiated under Project No. 7360, "Materials Analysis and Evaluation Techniques", Task No. 73605, "Design and Evaluation Data for Structural Metals", formerly RDO No. 614-13, "Design and Evaluation Data for Structural Metals", and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. A. W. Brisbane acting as project engineer.

This report covers work performed from March 1954 to November 1954.

ABSTRACT

In Part I data on the hydrogen embrittlement of one heat of 4340 steel are presented and evaluated. The data represent the mechanical behavior of the embrittled steel in the presence of stress-concentrations. Two different methods of embrittling the steel were employed. The first was to cathodically embrittle the specimens in a bath of 10 percent NaOH, and the second was to introduce embrittlement through the commercial plating of the specimens.

Three different tests were performed to evaluate the effect of hydrogen embrittlement on the steel under different test conditions. The first was a tensile test and the second a bend test. In both of these tests several loading rates were used to evaluate the effect of embrittlement under different loading conditions. The third test performed was a stress-rupture test to render the evaluation of embrittlement under sustained-load applications possible.

Several of the tests examined are suitable for the evaluation of hydrogen embrittlement. However, the bend test is the simplest, and most economical of these tests. At the same time it is a sensitive method of evaluating hydrogen embrittlement in ultra-high-strength steels.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. Whitmore
Technical Director
Materials Laboratory
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INTRODUCTION

The occurrence of delayed failure in steels heat treated to high strength levels is now generally attributed to the action of hydrogen introduced into the steel during electroplating, (1) (2). Because of this adverse effect of absorbed hydrogen on the strength and ductility of steel and, in particular, of high-strength steels, it is desirable that a convenient test be available for the evaluation of the brittleness induced by the presence of hydrogen.

Numerous test methods have been used in the past for the determination of brittleness in electroplated parts, namely, tension (1) (2), notch-tension (1) (2), impact (1), stress-rupture (2) (3), and bend tests (1) (4). Several of these tests, while informative, are expensive and, therefore, not suitable for routine testing. Other tests are insensitive and will not clearly indicate embrittlement due to hydrogen. Notable among the latter is the impact test, which does not respond to hydrogen, as will be shown later. Furthermore, for none of the tests which have recently been used in measuring hydrogen embrittlement has there been suitable correlation with the embrittlement that arises in the plating operation.

In the present report data for several different test conditions are presented in order to enable the evaluation of the hydrogen embrittlement of a steel heat treated to high strength level, on the one hand, and the suitability and sensitivity of the test methods, on the other hand. A limited number of test results on the recovery behavior of hydrogen-embrittled steels are also presented.

EXPERIMENTAL PROCEDURE

Material:

The 4340 steel studied was obtained as 9/16 in.dia. bar stock having the following chemical composition:

C	Mn	P	S	Si	Ni	Cr	Mo
0.38	0.71	0.017	0.02	0.30	1.90	0.96	0.24

Test Specimens:

All specimens used in this investigation were rough machined with a 0.020 in. oversize for grinding purposes after heat treatment. The bend, tensile, notch tensile and sustained-load specimens used are illustrated in Figure 1.

Heat Treatment:

Specimens were heat treated at 1600°F in a graphite block to minimize decarburization, oil quenched and tempered at temperatures ranging between 400°F and 800°F for one hour. Figure 2 shows the hardness of the 4340 steel as a function of tempering temperature.

Loading Rate Control:

Tension, notch-tension and bend tests were performed with a 60,000 pound universal testing machine. All loading rates reported are approximate and are referenced to the no-load rate of cross-head movement. The three rates used in the tension and notch-tension tests are as follows:

a) High Rate: 2 in. per min. = approximately 2500 lbs. per sec.

b) Medium Rate: 0.2 " " = approximately 250-400 " " "

c) Low Rate: 0.02 " " = approximately 35-40 " " "

In addition to the above three rates, impact rates were employed in the bend test by the use of a standard impact testing machine.

Constant-load tests were conducted in stress-rupture test machines in order to simulate sustained-load applications.

In all tests where concentricity of load was required suitable grips were employed. Figure 3 shows the concentric fixture used for all tensile investigations and Figure 4 the rig that was used for the bend tests.

Hydrogen-Impregnation Control:

Hydrogen was introduced into the specimen either by cathodic treatment in an electrolyte of 10 percent sodium hydroxide or, in a few tests by plating according to commercial practice.

In these tests sodium hydroxide was used to prevent etching of the specimen. This is believed to be especially important for the sharply-notched specimens where even minor etching could possibly modify the stress-concentration effects.

Hydrogen Content:

Tests were to be conducted on specimens containing both medium and high hydrogen contents. In determining these two levels of embrittlement, that developed by copper plating has been taken as a standard for medium hydrogen content. High hydrogen content has been taken as that resulting from an exposure five times that required for medium hydrogen content under the same cell-plating conditions.

Aging Time:

The time interval between the charging and testing of the specimens was maintained approximately constant, at about 5 minutes. However, to minimize diffusion of hydrogen from the specimen to the surroundings during the 5 minute interval, the specimens were placed in a solution of dry ice and acetone at approximately -80°C.

Plating:

A few bend tests were performed on commercially plated specimens. The various plating conditions were as follows:

a) Copper Plating
The solution consisted of:

Copper cyanide = 7.2 oz. per gallon
Sodium cyanide = 9.0 oz. per gallon
Potassium hydroxide = 1.0 oz. per gallon

and was maintained at 140°F. The current density used was 20 amp. per sq. ft.
Plate thickness = 0.0005

b) Cadmium Plating
The solution consisted of:

Cadmium metal = 3.0 oz. per gallon
Sodium hydroxide = 2.6 oz. per gallon
Sodium cyanide = 16.4 oz. per gallon
and was maintained at room temperature. The current density used was 30 amp. per. sq. ft.
Plate thickness = 0.00025-0.0003 in.

c) Chromium Plating
The solution consisted of:

Chromic acid = 29.0 oz. per gallon
Sulphuric acid = 0.2 oz. per gallon
and was maintained at 130°F. The current density used was 2 amp. per sq. in.
Plate thickness = 0.0003-0.0005 in.

The copper plating conditions employed in the sustained-load tests were as follows:

The solution consisted of:

Sodium hydroxide = 0.00374 gm. per c.c.
Copper cyanide = 0.0262 gm. per c.c.
Rochelle salt = 0.03 gm. per c.c.
and was maintained at 120°F. The current density used was 100 ma. per sq. in. for a period of 10 minutes.

EXPERIMENTAL RESULTS

Bend Tests:

Preliminary experimentation served to evaluate the effects of medium and high hydrogen contents. For this purpose bend tests were used.

The results of bend tests on smooth and notched specimens are presented in Figures 5a, 5b and 5c. The ductility of smooth specimens measured by the deflection at fracturing clearly revealed the effects of various conditions of embrittlement, and various loading rates, Figure 5a. The fracture load follows a trend similar to that of ductility but it is more difficult to determine accurately and is less sensitive, Figure 5b. The ductility of notched specimens was found to be rather insensitive, and this also applies to the fracture load, but to a less degree, Figure 5 c. Because of this lack of sensitive response to hydrogen embrittlement exhibited by notched specimens, all the testing was performed on smooth specimens, Figures 5a and 5b, except for the one set of notched specimens presented in Figure 5c.

The most notable feature of Figure 5a is the pronounced dependence of hydrogen embrittlement on loading rate. Specimens embrittled under the most severe conditions remained ductile in the impact test. In contrast, a decrease in the loading rate by about two orders, to the maximum loading rate possible in an ordinary tensile machine, led to pronounced embrittlement under nearly all controllable cell-operating conditions. For cell-operating conditions which did not lead to gas evolution, no embrittlement was observed for any exposure time. For cell-exposure times of 20 minutes or more hydrogen embrittlement was observed with this test at current densities of 4.5 ma. per sq. in. or greater.

Bend-test data for plated specimens, Figure 6, indicate that hydrogen embrittlement due to plating is severe and recovery has not taken place after holding at room temperature for 48 hours. Thus, if plating conditions are to be simulated, rather severe cathodic treatment is required. For this reason two hydrogen contents have been defined in terms of current density taken at approximately 1/2 amp. per sq. in. for one-half hour exposure for medium hydrogen content, and for two and one-half hours exposure for high hydrogen content.

Additional data presented in Figure 6 pertain to the recovery from hydrogen embrittlement of plated specimens. Prolonged holding at room temperature leads to slight recovery after copper plating. Effective recovery, however, is not possible at room temperature within holding times of over a week, so that baking procedures are indicated.

Within the sensitivity limits of the bend test both copper- and cadmium-plated specimens are recovered after baking for 24 hours at 300°F*. The data in Figure 6 indicate that shorter baking times at this temperature are not fully effective. Longer times are not required by the evidence of this test. Full recovery of the chromium-plated specimens was not effected by any of the treatments used, although all baking treatments did lead to an improvement in ductility.

Thermal cycling treatments have been reported as being potentially effective for the elimination of hydrogen, (5). For some applications such cycling has potential advantages. The few data presented in Figure 6 indicate that thermal cycling has no advantage over heating at a constant temperature in eliminating hydrogen embrittlement.

Tension and Notch-Tension Tests:

Tension tests on smooth specimens yielded, according to Figures 7 and 8, complex behavior due to the stress-concentration effect at the fillet under the head. Therefore, these results will be discussed further below.

The many combinations of pertinent factors involved in the tension and notch-tension investigation require a large volume of experimental work in order to accurately establish their effects. As large scattering has been observed in this type of test on hydrogen embrittled specimens, establishing probable trend curves requires special efforts. To arrive at such trend curves, it is believed that the results of the large total volume of work can be used to compensate for the lack of sufficient parallel tests in each individual test condition. The procedure used consisted of first averaging all values for a particular strength level and thus establishing a general average trend curve for the effect of strength level. It was then observed that for approximately one half of the individual test series the experimental points agreed within ± 10 percent to a trend curve nearly parallel to the average curve. In other instances only one point deviated to a greater extent from such trend curves, while in only a few instances rather large deviations were observed. This principle can be further utilized with respect to the other embrittling factors used in the investigation. The strength should decrease with decrease in the loading rate, or with increase of either hydrogen content or stress-concentration. It was discovered that trend curves derived as described above conformed to this principle. In contrast, nearly all points which deviated from the trend curves were also found to be in disagreement with the trend demanded by one or more of the factors which should reduce ductility.

*The notch properties of 4340 steel heat treated to very high strength level by tempering at 400 to 450°F are modified by prolonged holding in this temperature range. About 300°F is considered as the maximum safe temperature for baking times of 24 hours.

The final result of such an analysis was that all trend curves belonged to a single family. The location of an individual curve in such a family was determined by any single (accurate) test point. Identical trend curves frequently resulted from various combinations of the embrittling factors.

The tests on smooth specimens were unsuccessful in that failure of the high-strength conditions usually occurred at the fillet under the head. The stress-concentration at this location was rather low, approximately 1.7. It was only at 800°F tempering temperature that failure of the smooth specimens always occurred in the test section. Despite such premature failures, the test results reported in Figures 7 and 8 are very instructive. The competition between the two different areas of potential failure led to the conclusion that the rather mild condition of stress-concentration at the fillet caused a considerable higher reduction in strength than would be expected to result from the same stress-concentration value introduced by a circular notch. Its effect closely corresponded to that of a considerably sharper cylindrical notch introducing a stress-concentration between 3 and 5, Figures 15 and 16. The only possible explanation is the fact that the conditions existing at the fillet involve more factors than a simple stress-concentration effect. One of these is the existence of a hydrogen content higher than that present in a circular notch as discussed in a succeeding paragraph. A second is the existence of a stress-gradient different from that in a circular notch. Finally, there is the possibility of eccentric loading which is detrimental to the strength of embrittled specimens with a fillet. The results obtained for the smooth specimens are the product of all factors involved and the isolation of each effect is impossible with the limited data that are available. The stress-concentration calculated for the fillet is marked with a subscript (K_1), to differentiate it from the other type of stress concentration.

The introduction of hydrogen into a notched specimen, under all conditions studied, revealed according to Figures 9 to 16, severe embrittlement for the 400 to 700°F tempering temperatures and rather moderate embrittlement for the 800°F temperature.

As in the case of the smooth specimens, the strength decreases i.e. the trend curve is lowered, as the loading rate is decreased or as either the hydrogen content or the stress-concentration is increased.

In Figures 15 and 16 the effectiveness of both medium and high hydrogen contents in leading to embrittlement for the various stress-concentration conditions is particularly emphasized. Thus, a very sensitive index of hydrogen embrittlement is provided by the smooth specimens containing a notch of low stress-raising capacity at the juncture of the head and test section. This observation has highly-significant practical implications. However, it is difficult to account for it on the basis of theory.

It appears that the inversion of effect of stress-concentration in promoting embrittlement is due to a multiplicity of factors discussed previously. Among these is the change in the cell-operating conditions caused by the change in the geometry of the specimen which constitutes the cathode of the cell. For example, the "throwing power" of a plating cell is altered by the geometry of the cathode. As a consequence the hydrogen content of the metal at the base of a sharp circular notch would be less than that in a volume of metal in the smooth contour of a well filleted shoulder. Further, the diffusion for the sharp circular notch would be along a two-dimensional gradient while for the smooth specimen this gradient would be one-dimensional. It may be expected, therefore, that the hydrogen content of the shoulder of the smooth specimen would be higher and would remain higher than that at the base of the sharp circular notch.

Sustained-Load Failures:

Sustained-load failures have been one of the most perplexing types of failures traceable to the action of hydrogen. This type of failure has recently been studied at length at Case Institute of Technology (2). The embrittling conditions employed in the Case studies were probably mild compared to plating conditions and were very mild compared to those used here. Results obtained in the present program under the more severe embrittling and plating conditions for smooth and notched specimens are presented in Figures 17 to 26.

Although the test section of the smooth specimens used in this investigation for sustained-loads was reduced, see Figure 1, the specimens broke either under the head or at the 1/8 in. rad. fillet, where the stress-concentration is approximately 1.1 As in the case of the smooth tensile tests, the results for the smooth specimens in the stress-rupture investigations were evaluated on the basis of the stress-concentration existing at the locations of failure. Again as in the case of the tensile experiments, the comparatively mild notch under the specimen head caused a higher reduction in strength than would result from the same stress-concentration due to a circular notch. The results presented in Figures 22 and 23 show that the effects of a stress-concentration of 1.7 under the specimen head are comparable to those of a stress-concentration of 5 introduced by a circular notch. The only explanation available would be the same as was offered for the results of the tensile tests.

From Figures 17 to 26 it is seen that for all tests sustained loads led to reduced fracture strength for the embrittling and plating conditions studied.

The sustained-load test data are plotted on linear coordinates, Figures 25 and 26. In these latter two figures the transitional behavior emphasized in the semi-log plots is still observed, but it is seen to depend on the experimental conditions for its time-wise location.

The fracture sections in specimens broken by sustained loads are composed of a central circular section surrounded by an outer peripheral and usually symmetrical rim area, Figure 27. The low-load fractures, therefore, closely resemble low-load fatigue fractures. In the present work the development of the two fracture zones has been considered as being an analogy to the development of the two fracture zones in typical fatigue failures. Thus, through the combined action of high stress and hydrogen presence, a fracture is initiated. This crack is propagated as a function of the rate of hydrogen diffusion to the propagating crack front and as as a function of the rate of increase of the stress concentration and stress level due to the change in notch geometry through the development of the crack and due to reduction in the cross section through propagation of the crack. These factors may untimately lead to failure at nominal stresses very much lower than the failure stresses expected.

According to the above theory, the notch strength of the specimen referred to the instantaneous fracture section should remain constant after a stable notch condition has been reached, i.e. after a maximum stress concentration has been achieved. Data which may be examined in this way are presented in Figure 28.

The notch strength data fall into two groups in the first of which the notch strength remains constant throughout, and indicating that the steel in this condition is notch insensitive. In the second group the notch strength drops with increasing time to rupture and stabilizes at a value of about 125,000 psi. The circumstances of the test would indicate that this reduction in the instantaneous notch strength is due to the increase in the notch sharpness and thus to the increase in the stress concentration due to the crack. This reduction is probably not due to the action of the hydrogen on the structure as indicated by the results for the steel tempered at 800°F.

The cracks postulated from examination of Figure 28 should be detectible in the early stages of the test, and the photographs shown in Figure 29 were taken at two stages of crack development in a notched copper plated specimen under different loading conditions. The crack is very fine and is difficult to photograph, but is easily observable when the specimen under load is examined with a high power lens.

The extent of crack penetration for the specimens for two stress concentrations and three tempering temperatures was measured as a function of time to fracture, Figure 30. Of interest is the initial rapid rate of growth of the crack in the specimens tempered at 500 and 700°F as compared to the crack growth in specimens tempered at 800°F. The rates of continued growth in the three sets of specimens after the preliminary stage appear to be constant. This crack growth, as explained earlier, is due to hydrogen diffusion and these data indicate that the rate of hydrogen diffusion for the three steel conditions is effectively constant.

The differences in the initial crack growth rates are possibly due to one or both of two factors, namely, a) structural differences resulting from the different tempering treatments for the different steel conditions, and, b) reduction in the hydrogen content at the base of the sharper notch to be attributed to the cell-operating conditions as discussed earlier.

The depths of hydrogen penetration-time data inferred from Figure 30 are in agreement with the data presented by Case (2).

DISCUSSION

The experimental data that have been presented here indicate that a variety of tests are available for evaluating hydrogen embrittlement in ultra-high-strength steels. Unquestionably the simplest test method examined is the bend test of smooth specimens. Low cost and ease of testing are important factors which may be advanced in favor of this test procedure.

The qualitative features of the data for cathodic embrittlement in caustic soda are in essential agreement with the data of Case (2) and Battelle Memorial Institute (3). The present data quantitatively represent an embrittlement condition intermediate between the extreme conditions studied by the other two groups of investigators. The use of the non-etching cell environment, therefore, does not lead to qualitative differences in the data. The data obtained for plated specimens, however, indicate qualitative differences from cathodically-embrittled specimens. These differences appear mainly in the recovery characteristics of the embrittled and plated specimens. For example, Case reports considerable recovery of embrittled specimens on holding at room temperature for 24 hours. Similar results have been reported by Syracuse University (6) for holding temperatures of about 80, 32 and -100°F for bend specimens. (The time for full recovery at each temperature varied widely in this study.) On the other hand, plated specimens held for more than a week at room temperature are still fully as brittle as freshly embrittled specimens or are only slightly recovered. Furthermore, while certain plated conditions can be restored through baking to a practically unembrittled condition there is a question whether this is possible after chromium plating. It is believed that these different behaviors result from the different cell conditions under which hydrogen is introduced into the steel and from the different diffusion systems present in the embrittled specimens after removal from the electrolytic cell.

In the cathodic treatment of the specimen, hydrogen may be considered as being plated on the specimen surface. This surface is an efficient catalyzer of the reaction $H_{(ionic)} \rightarrow H_{(molecular)}$ under this plating condition. Little of the hydrogen passed through the cell enters the specimen. This condition is modified at least in part during plating and the hydrogen that is plated on during the true plating operation is retained on the surface and is free to diffuse into the steel from this location. Some measure of the hydrogen entrapped in the plate is given by the plating efficiency of the plating bath used. While side reactions have been neglected here in determining cell efficiencies, there is little question that large amounts of hydrogen are present in the various plates. Once the plated and cathodically

embrittled specimens are removed from the electrolytic cells physically different hydrogen gradients are established at the steel surface for the two conditions. These two types of gradients are indicated in Figure 31.

For the cathodically embrittled specimen the surface is open and hydrogen is free to diffuse immediately away. While the hydrogen content at the surface is thus reduced, an appreciable fraction of the total hydrogen in the specimen is also probably lost in a very short time. This is contrary to the condition which is visualized for the plated specimen where the hydrogen content at the plate-specimen interface will remain at a high level until it is dissipated by diffusion either into the specimen or through the plate, to be oxidized or otherwise removed from the plate surface. The diffusion process, however, may be complex and may lead to a stationary state with high hydrogen content at the plate-steel interface, in which case a nearly permanent state of hydrogen embrittlement would result. Under steady load application this hydrogen would then be available for the development of a crack and ultimate fracture as has been described.

Many questions have been raised in the examination of the problem of hydrogen embrittlement. One of these, for example, is the role of hydrogen in promoting crack formation. Another is the possibility that certain plating operations may lead to permanent damage and embrittlement of the steel. Finally, the coating of plated specimens may represent a source of hydrogen leading to embrittlement of the steel. These questions must be left open at the present time.

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APPENDIX

Tables I - III give the results of all tests performed in this investigation.

Table I	Bend Data
Table II	Tensile Data
Table III	Sustained-Load Data

TABLE I (a)

Results of Hydrogen Embrittled Bend Test Specimens of 4340 Steel (1600°F/oil)
Test Temp. = R.T.

Type of Spec. and Temp. Temp.	Current Density ma.per sq.in.	Charging Time min.	Strain Rate in.per min.	Deflection in.	Fracture Load-lb.
Notched 700°F	447	0	0.02	0.029	8,700
	"	45	"	0.022	5,800
	"	90	"	0.017	5,400
	"	135	"	0.018	5,700
Smooth 400°F	10	5	0.20	0.210	15,500
	"	10	"	0.150	13,700
	"	15	"	0.179	14,000
	"	20	"	0.142	13,500
	"	30	"	0.095	12,600
	"	60	"	0.082	11,900
	"	90	"	0.065	11,500
	"	120	"	0.054	10,900
	200	5	"	0.140	13,500
	"	15	"	0.104	12,400
	"	30	"	0.062	11,400
	"	60	"	0.060	11,400
	"	90	"	0.075	11,700
	"	120	"	0.067	11,700
	10	2-1/2	2.00	0.260	17,600
	"	5	"	0.200	13,800

TABLE I (a) Cont'd.

Results of Hydrogen Embrittled Bend Test Specimens of 4340 Steel (1600°F/oil)
Test Temp. = R.T.

Type of Spec. and Temp. Temp.	Current Density ma.per sq.in.	Charging Time min.	Strain Rate in.per min.	Deflection in.	Fracture Load-lb.
	10	10	2.00	0.200	14,000
	"	10	"	0.240	18,800
	"	15	"	0.190	15,700
	"	30	"	0.160	13,300
	"	60	"	0.125	12,600
	"	90	"	0.114	12,700
	"	90	"	0.095	12,200
	"	120	"	0.100	12,300
	200	5	"	0.140	13,500
Smooth	"	15	"	0.155	14,900
400°F	"	30	"	0.170	13,500
	"	60	"	0.136	14,200
	"	90	"	0.130	12,600
	"	90	"	0.120	13,800
	"	120	"	0.106	14,600
	0	0	Impact	0.360*	
	10	5	"	0.360*	
	"	20	"	0.360*	
	200	60	"	0.360*	
	1000	120	"	0.360*	

*Did not fracture

TABLE I (b)

Results of Commercially Plated Bend Test Specimens of 4340 Steel (1600°F/oil), Tempered at 400°F. Test Temperature = R.T.

Type of Plating	Treatment given to Specimen	Strain Rate in.per min.	Deflection in.	Fracture Load-lb.
Cadmium Plating	Aged for48 hrs. after Plating	0.20	0.095	11,600
	Aged for 212 hrs. after Plating	"	0.095	12,200
	Heated to 300°F for 10 hrs.	"	0.250*	
	Heated to 300°F for 24 hrs.	"	0.260*	
	Cycled 20 times (R.T.-300°F)	"	0.100	12,100
Chromium Plating	Aged for 48 hrs. after Plating	"	0.100	12,000
	Aged for 212 hrs. after Plating	"	0.100	12,200
	Heated to 300°F for 10 hrs.	"	0.215	14,100
	Heated to 300°F for 24 hrs.	"	0.195	13,700
	Cycled 20 times (R.T.-300°F)	"	0.195	14,300
Copper Plating	Aged for 48 hrs. After Plating	"	0.100	13,000
	Aged for 212 hrs. After Plating	"	0.165	13,700
	Heated to 300°F for 10 hrs.	"	0.200	14,100
	Heated to 300°F for 24 hrs.	"	0.265*	
	Cycled 20 times (R.T.-300°F)	"	0.190	13,700

*Did not Fracture

TABLE II (a)

Results of 0.3 in.dia. Tensile Specimens of 4340 Steel (1600°F-oil) Hydrogen Embrittled in a Bath of 10 % NaOH at 30°C with a Current Density of 447 ma.per sq.in. for 1/2 hr. and Tested at a Loading Rate of 0.02 in. per min. Test Temp. = R.T.

Tempering Temp.-°F	Strength - 1000 psi				Percent Reduction of Area			
	K = 1	K = 3	K = 5	K = 10	K = 1	K = 3	K = 5	K = 10
400	232.6	188.2	178.1	205.5	*	0.6	1.4	0.9
500	170.7	233.7	174.1	149.7	*	1.4	1.1	0.6
600	240.1	259.8	210.7	156.9	49.1	0.3	0.8	0.8
700	226.3	229.7	180.0	202.0	50.8	1.7	1.1	0.6
800	202.1	272.0	260.6	271.0	49.9	4.2	2.0	3.7

*Broke under head

TABLE II (b)

Results of 0.3 in.dia. Tensile Specimens of 4340 Steel (1600°F-oil) Hydrogen Embrittled in a Bath of 10% NaOH at 30°C with a Current Density of 447 ma. per sq. in. for 1/2 hr. and Tested at a Loading Rate of 0.20 in. per min. Test Temp. = R. T.

Tempering Temp.-°F	Strength-1000 psi				Percent Reduction of Area			
	K = 1	K = 3	K = 5	K = 10	K = 1	K = 3	K = 5	K = 10
400	213.6	233.7	173.8	192.4	*	2.3	1.4	1.7
500	255.3	266.9	203.3	168.0	*	1.1	1.6	0.8
600	213.5	249.3	208.8	184.7	*	2.0	1.7	1.7
700	223.0	239.5	201.9	237.1	50.7	1.1	1.1	2.3
800	200.1	274.1	262.6	275.9	53.5	2.6	4.5	6.2

*Broke under head

TABLE II (c)

Results of 0.3 in. dia. Tensile Specimens of 4340 Steel (1600°F/oil) Hydrogen Embrittled in a Bath of 10% NaOH at 30°C with a Current Density of 447 Ma. per sq. in. for 1/2 hr. and Tested at a Loading Rate of 2.00 in. per min. Test Temp. = R. T.

Tempering Temp.-°F	Strength - 1000 psi				Percent Reduction of Area			
	K = 1	K = 3	K = 5	K = 10	K = 1	K = 3	K = 5	K = 10
400	268.6	247.1	221.1	225.9	50.8	2.3	1.2	0.6
500	245.8	283.0	237.1	185.0	*	2.9	2.7	0.8
600	216.2	236.6	190.7	168.0	*	1.7	1.1	1.4
700	222.8	250.0	---	200.0	45.5	3.1	---	3.2
800	215.7	267.7	260.6	247.0	52.2	5.4	3.1	5.5

*Broke under head

TABLE II (d)

Results of 0.3 in. dia. Tensile Specimens of 4340 Steel (1600°F/oil) Hydrogen Embrittled in a Bath of 10% NaOH at 30°C with a Current Density of 447 Ma. per sq. in. for 2-1/2 hrs. and Tested at Loading Rate of 0.02 in. per min. Test Temp. = R. T.

Tempering Temp.-°F	Strength - 1000 psi				Percent Reduction of Area			
	K = 1	K = 3	K = 5	K = 10	K = 1	K = 3	K = 5	K = 10
400	254.6	292.4	252.1	232.4	*	1.7	2.3	0.9
500	280.1	295.2	247.1	210.8	35.6	3.4	1.7	1.4
600	221.4	262.0	232.9	190.3	*	2.3	1.1	1.7
700	230.4	256.3	245.0	213.7	47.5	3.6	2.3	1.4
800	204.1	292.0	284.5	267.6	50.5	6.0	4.5	3.1

*Broke under head

TABLE II (e)

Results of 0.3 in. dia. Tensile Specimens of 4340 Steel (1600°F/oil) Hydrogen Embrittled in a Bath of 10% NaOH at 30°C with a Current Density of 447 ma. per sq. in. for 2-1/2 hrs. and Tested at a Loading Rate of 0.20 in. per min. Test Temp. = R. T.

Tempering Temp.-°F	Strength - 1000 psi				Percent Reduction of Area			
	K = 1	K = 3	K = 5	K = 10	K = 1	K = 3	K = 5	K = 10
400	---	188.2	125.7	157.1	---	0.8	0.3	0.0
500	147.8	180.3	191.1	120.1	*	1.7	0.6	0.6
600	141.2	163.8	131.4	147.3	*	2.8	0.9	0.3
700	133.0	179.3	159.5	140.8	*	1.4	0.6	0.6
800	198.9	214.1	223.0	271.0	49.8	2.3	2.8	3.1

*Broke under head

TABLE II (f)

Results of 0.3 in. dia. Tensile Specimens of 4340 Steel (1600°F-oil) Hydrogen Embrittled in a Bath of 10% NaOH at 30°C with a Current Density of 447 ma. per sq. in. for 2-1/2 hrs. and Tested at a Loading Rate of 2.00 in. per min. Test Temp. = R. T.

Tempering Temp.-°F	Strength - 1000 psi				Percent Reduction of Area			
	K = 1	K = 3	K = 5	K = 10	K = 1	K = 3	K = 5	K = 10
400	188.8	215.3	167.6	188.9	*	2.8	0.3	0.6
500	186.3	246.4	160.0	149.7	*	0.6	1.1	0.6
600	153.9	185.4	150.9	147.7	*	0.8	0.0	0.3
700	173.8	167.4	169.5	180.3	*	0.6	0.0	2.0
800	203.1	255.7	230.0	276.4	52.8	4.8	2.3	5.5

*Broke under head

TABLE III (a)

Results of Sustained-Load and Constant Rate Tests on Hydrogen Embrittled Specimens from 4340 Steel (1600°F/oil) Test Temp. = R.T. Bath: 10% NaOH at 30°C-447 ma. per sq.in. for 1/2 hr.

Tempering Temp.	Type of Test	Stress-1000 psi		Time to Rupture-hrs.		Stress Based on Final Fracture Area-1000 psi
		K = 1	K = 5	K = 1	K = 5	K = 5
500°F (270,000 psi)	Stress Rupture Test	125.0	58.0	100→	114→	----
		155.0	75.0	120→	1.4	127.0
		190.0	102.0	0.6*	0.1	201.0
		170.0	150.0	0.6*	0.003	150.0
		150.0	199.0	0.2*	0	200.0
		202.0	---	0.1*	---	---
		95.0	---	0.05+	---	---
		98.0	---	0.03+	---	---
	Constant Rate Test	170.7	125.0	0.085+	0.045	---
		255.3	140.0	0.014+	0.0055	---
		280.1	180.0	0.003	0.0015	---
		---	203.0	---	0.0008	---
700°F (230,000 psi)	Stress Rupture Test	194.0	50.0	100→	165→	---
		89.0	65.0	19.9+	4.7	126.0
		94.0	77.0	19.0+	3.4	127.0
		176.0	162.0	13.2+	3.0	250.0
		230.0	185.0	1.5*	2.6	286.0
		240.0	108.0	0	2.1	181.0
		200.0	145.0	[118]→	0.4	233.0
		210.0	206.0	[3.3]*	0	273.0
		220.0	193.0	[1.5]*	0	240.0
		240.0	---	[0.01]	---	---
	Constant Rate Test	226.3	180.0	0.085	0.036	---
		223.0	201.0	0.014	0.0055	---
		230.0	245.0	0.002	0.0008	---

*Broke at Fillet
+Broke Under Head
[ ]Copper Plated
→ Did not Fracture

TABLE III (b)

Results of Sustained-Load and Constant Rate Tests on Hydrogen Embrittled and Copper Plated Specimens from 4340 Steel (1600°F/oil) Test Temp. = R. T.

Tempering Temp.	Treatment Given to Specimen	Type of Test	Stress - 1000 psi	Time to Rupture-hrs.	Stress Based on Final Fracture Area-1000 psi
			K = 10	K = 10	K = 10
800°F (210,000 psi)	Hydrogen Embrittled 447 ma.per sq. in.for 1/2 hr.	Stress Rupture Test	148.0	117 →	---
			179.0	308 →	---
			200.0	31	281.0
			237.0	15	309.0
			250.0	4.8	294.0
			276.0	0.008	310.0
		Constant Rate Test	271.0	0.1	---
			276.0	0.01	---
			268.0	0.001	---
	Copper Plated	Stress Rupture Test	187.0	100 →	---
			228.0	5.7	333.0
			201.0	5.2	280.0
			250.0	4.25	315.0
			259.0	1.1	295.0
			266.0	0.2	---

→ Did not Fracture

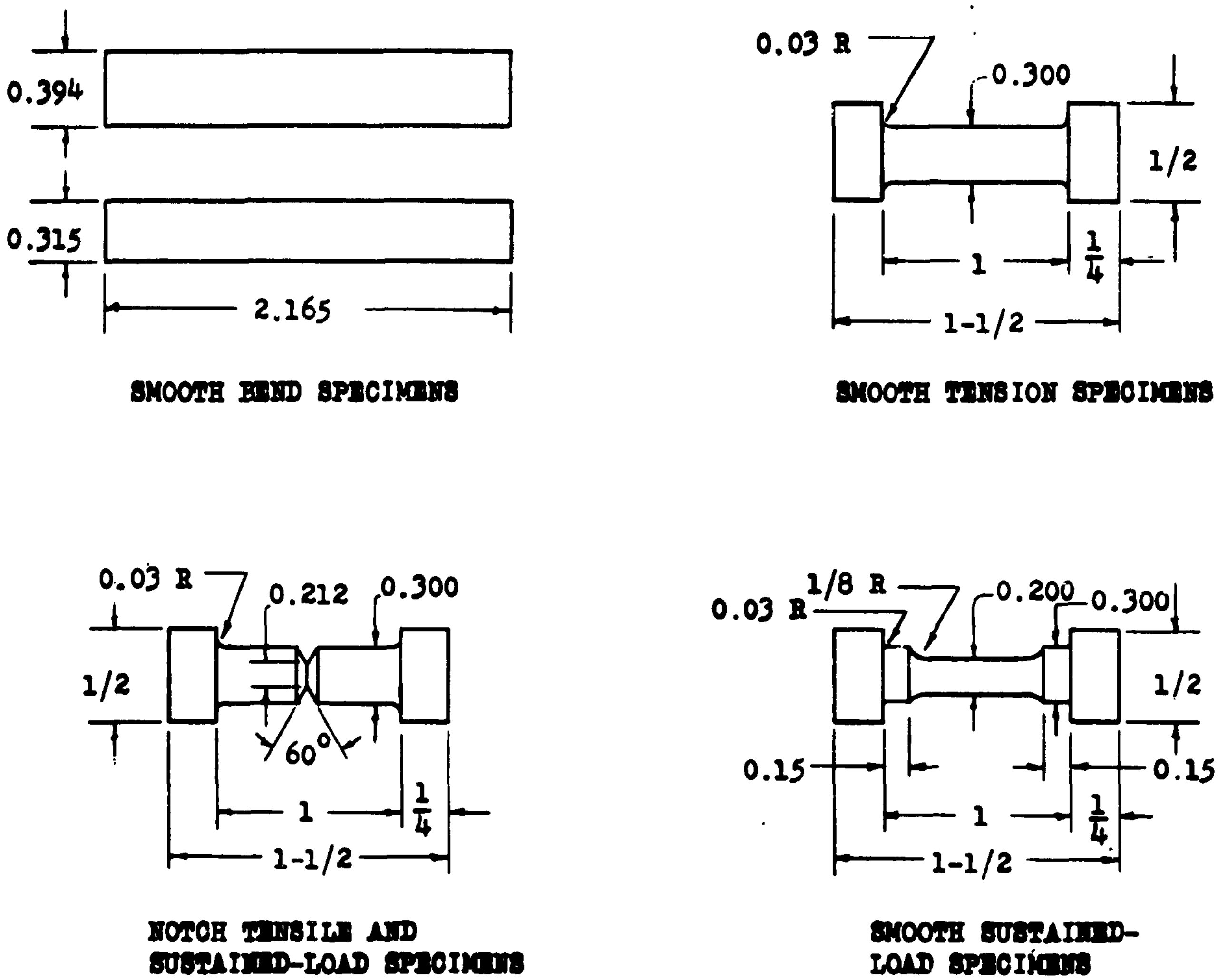


FIG. 1

TENSILE, SUSTAINED LOAD AND BEND SPECIMENS

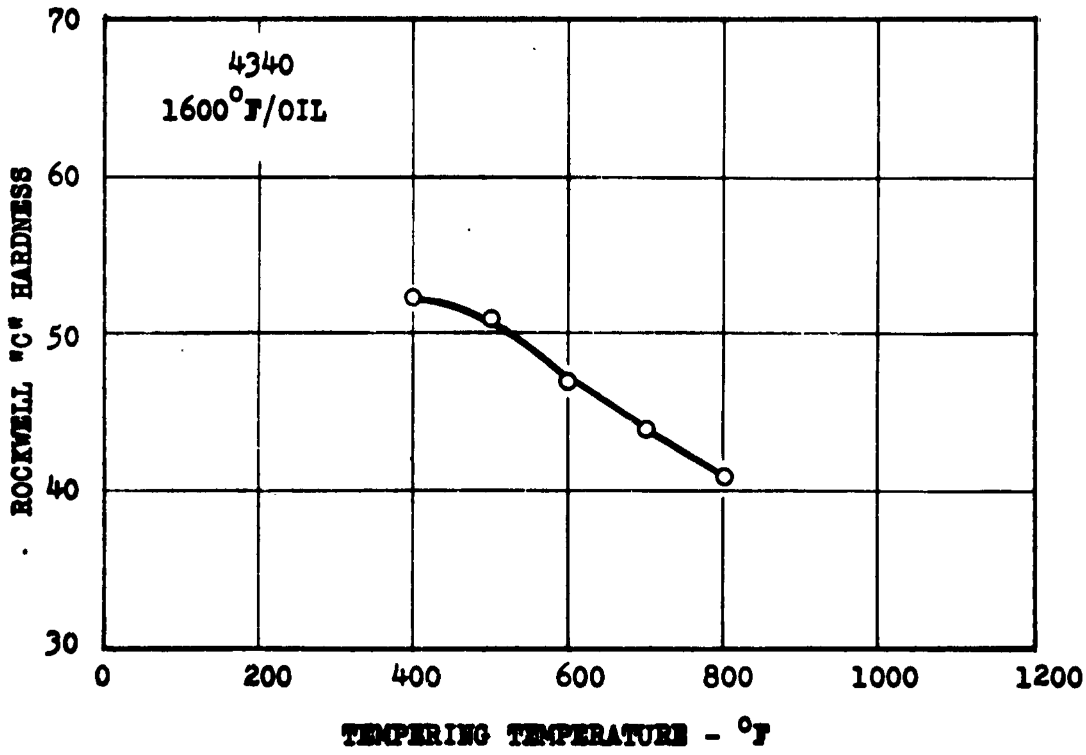


FIG. 2 HARDNESS AS A FUNCTION OF TEMPERING TEMPERATURE FOR THE 4340 STEEL STUDIED.

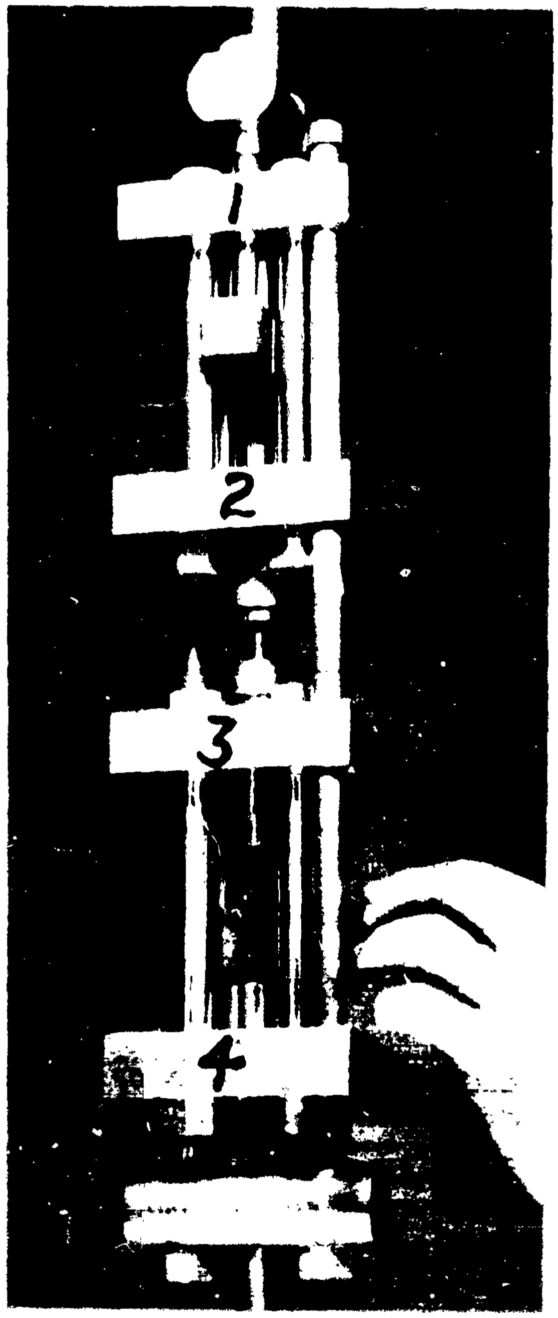


FIG. 3 FIXTURE FOR CONCENTRIC LOADING OF 0.3-IN. DIAMETER TENSILE SPECIMENS.

FIG. 4 FIXTURE USED FOR BEND TESTS.

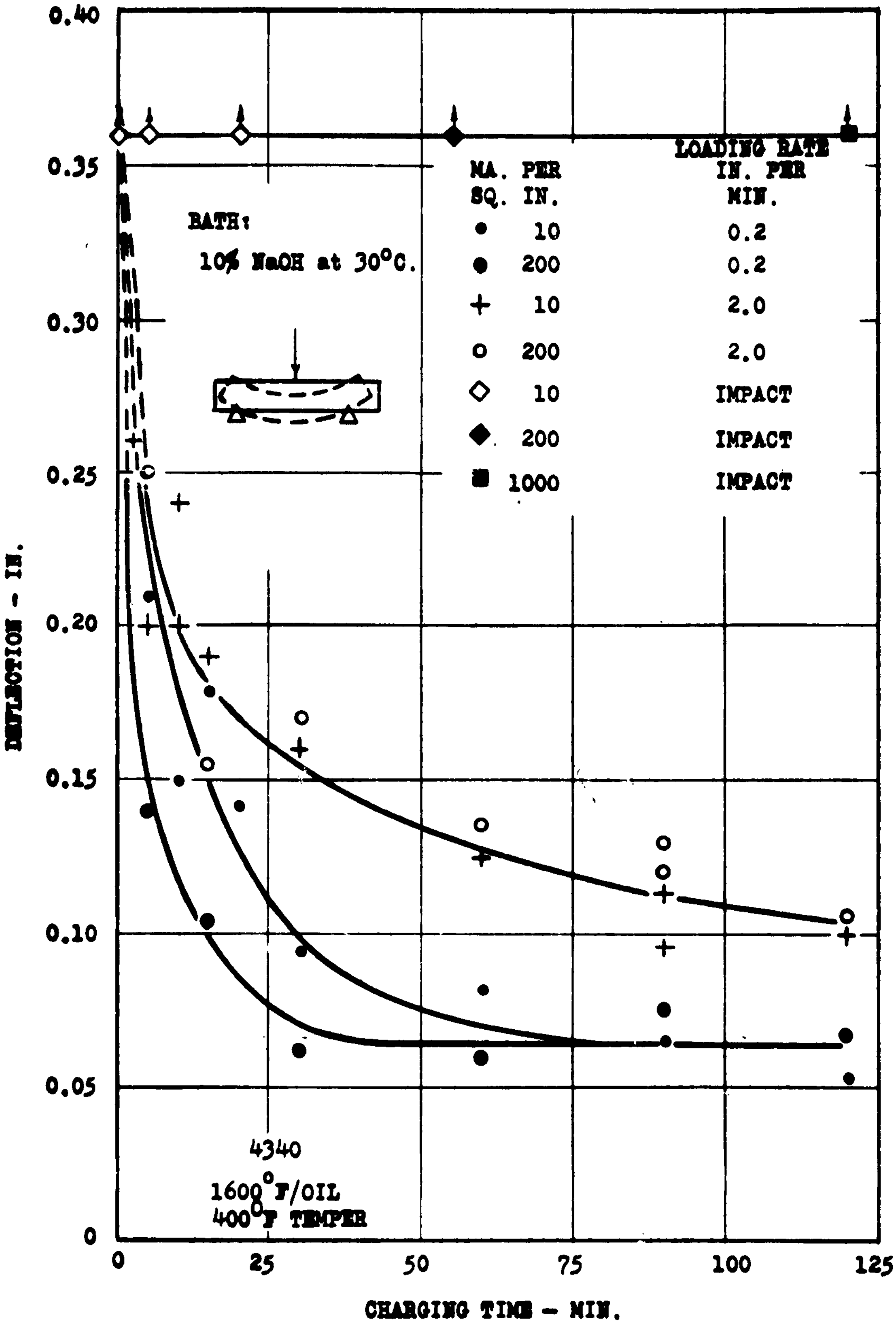


FIG. 5a DEFLECTION AS A FUNCTION OF CHARGING TIME FOR SMOOTH BEND SPECIMENS OF 4340 STEEL WITH STRAIN RATE AND HYDROGEN CONTENT AS PARAMETERS. TEST TEMP: R.T.

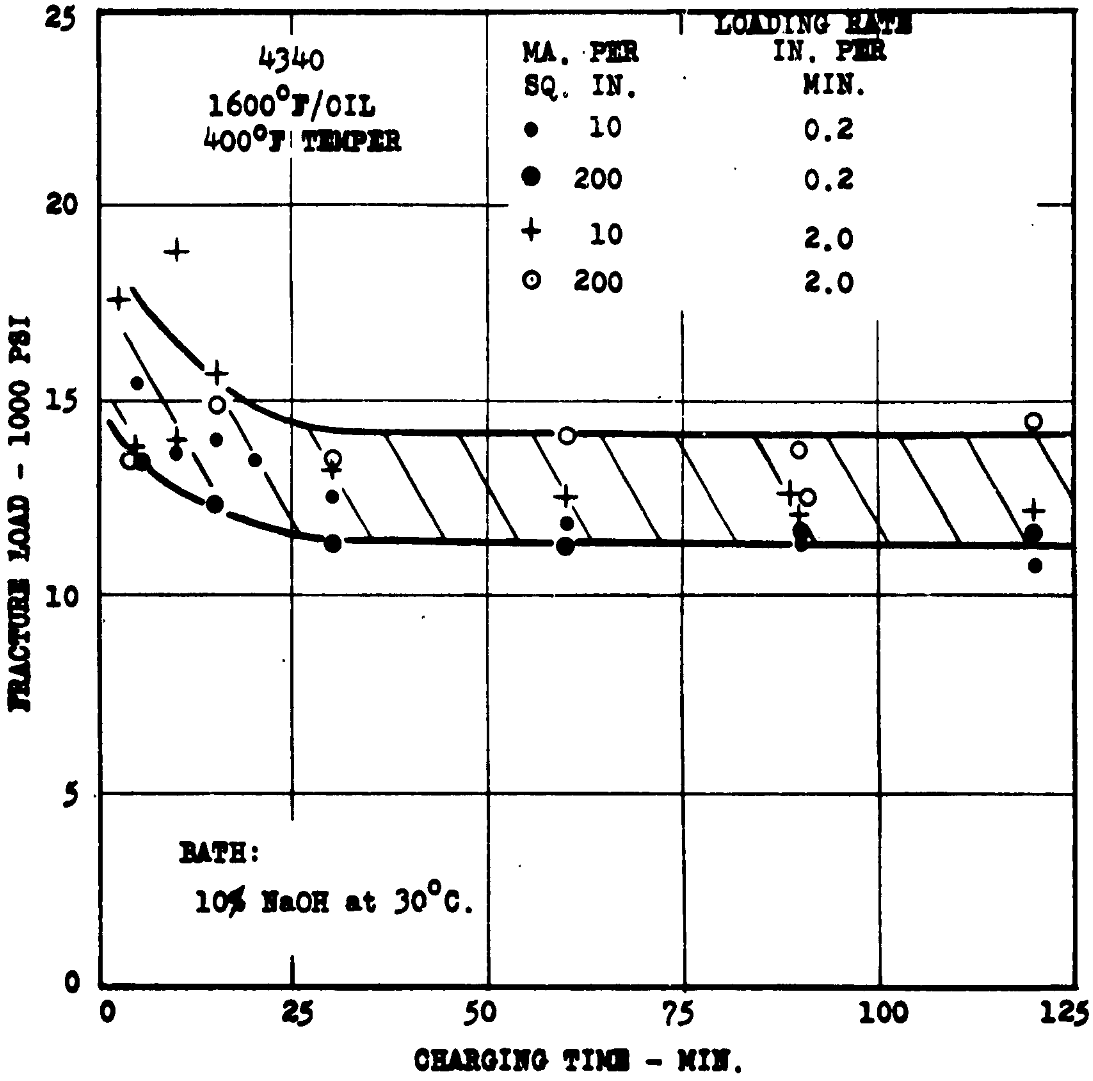


FIG. 5b FRACTURE LOAD AS A FUNCTION OF CHARGING TIME FOR SMOOTH BEND SPECIMENS OF 4340 STEEL WITH STRAIN RATE AND HYDROGEN CONCENTRATION AS PARAMETERS. TEST TEMP: R.T.

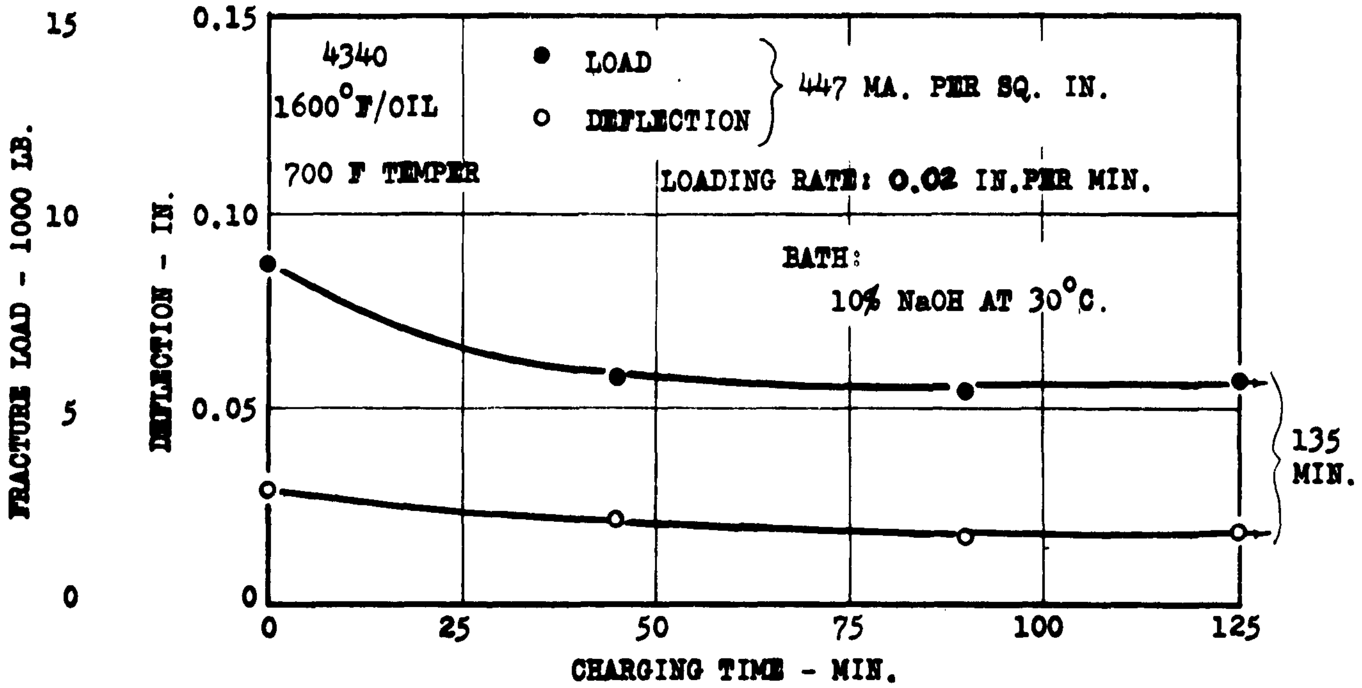


FIG. 5c DEFLECTION AND FRACTURE LOAD AS A FUNCTION OF CHARGING TIME FOR NOTCHED BEND SPECIMENS OF 4340 STEEL.
TEST TEMP: R.T.

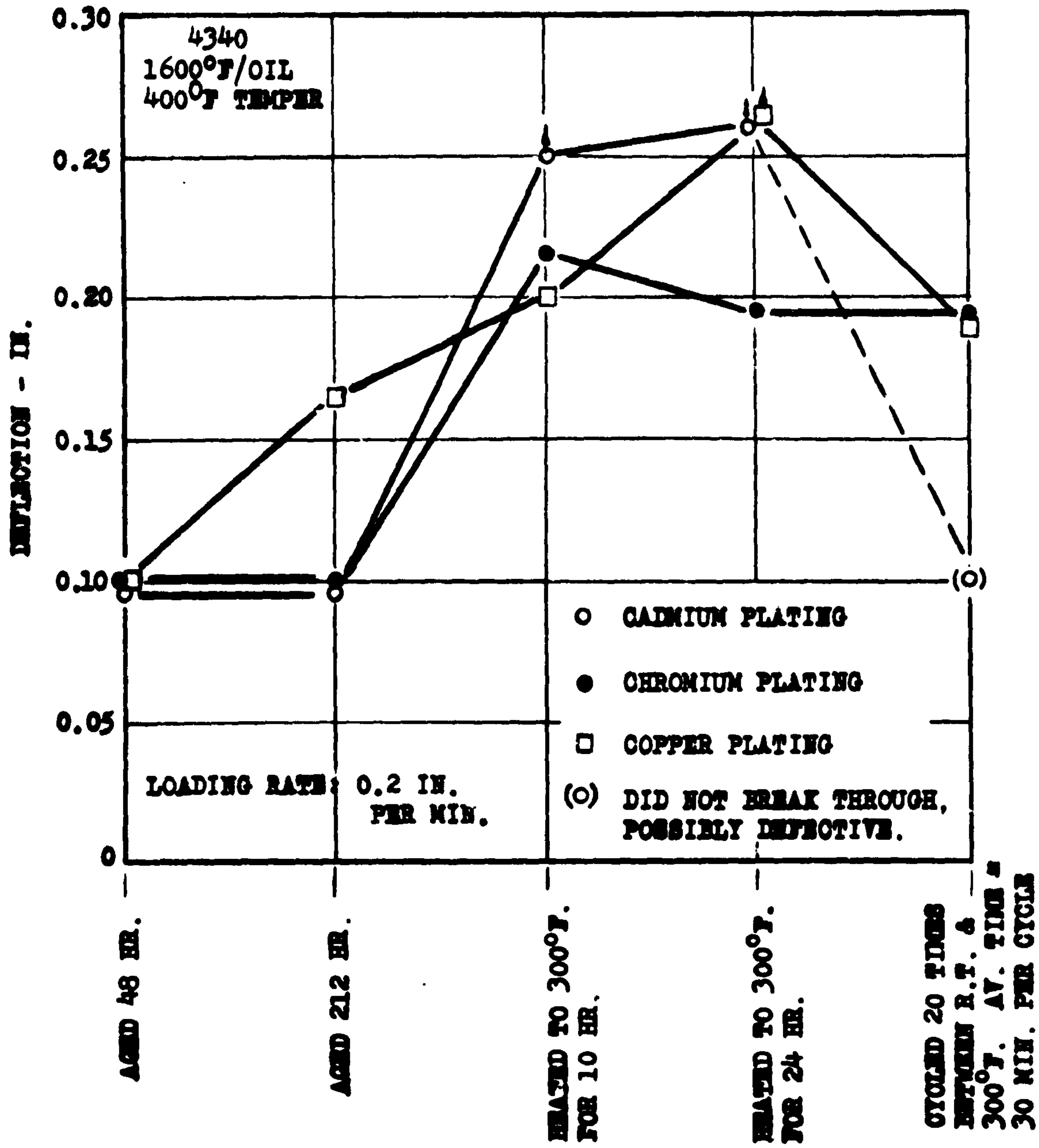


FIG. 6 EFFECT OF VARIOUS TREATMENTS ON THE DUCTILITY OF COMMERCIALLY PLATED "BEND" SPECIMENS OF 4340 STEEL. TEST TEMP: R.T.

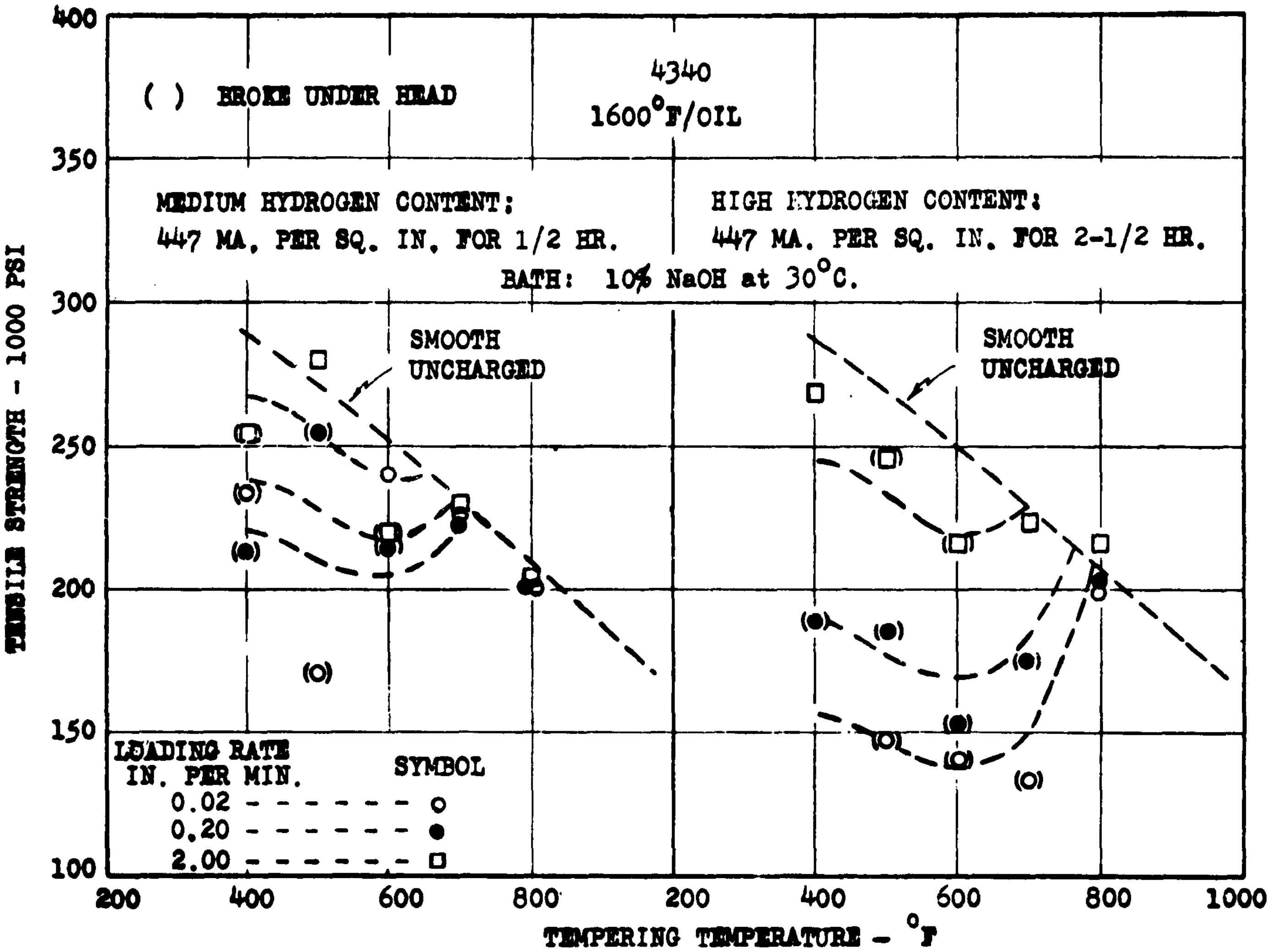
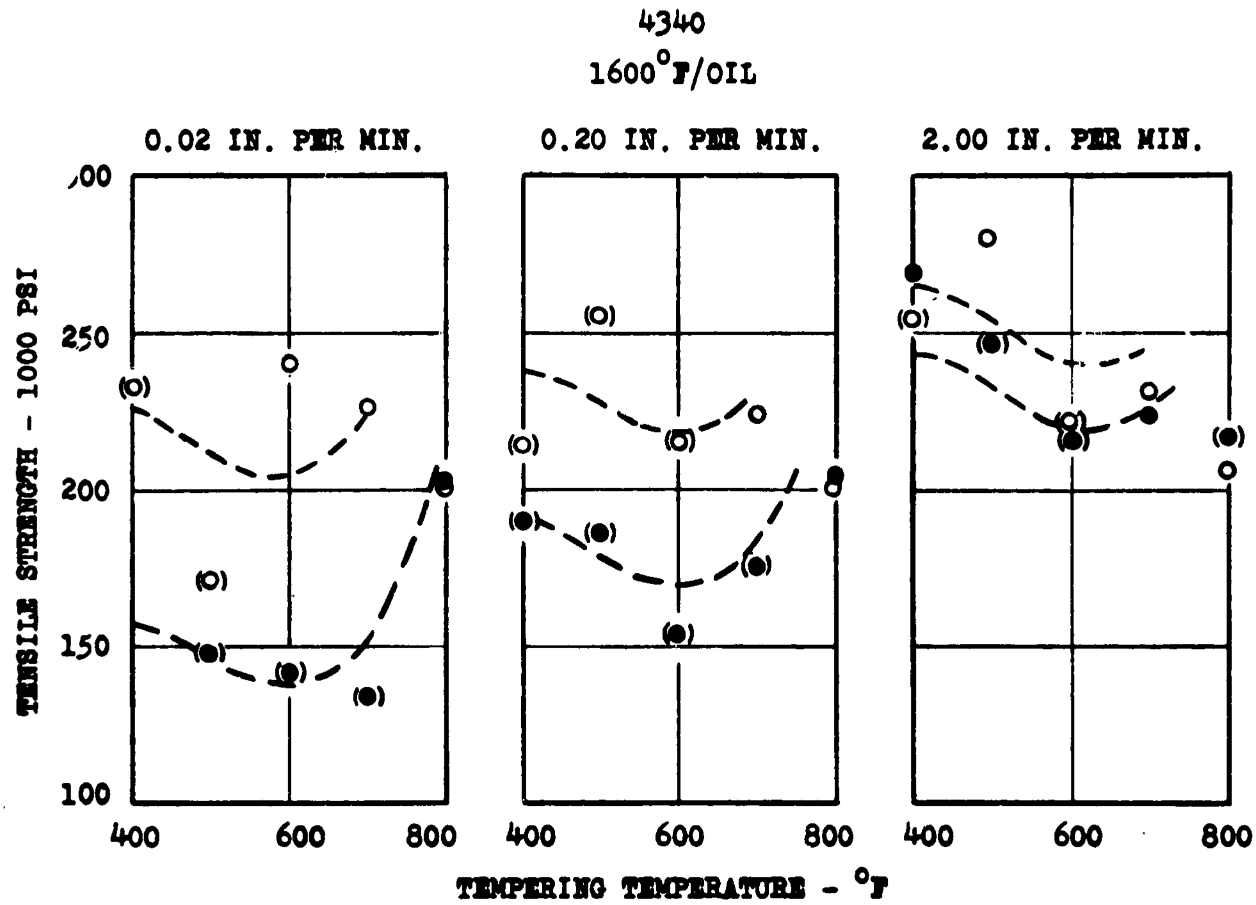


FIG. 7 TENSILE STRENGTH OF HYDROGEN EMBRITTLED 0.3 IN. DIA. TENSILE SPECIMENS OF 4340 STEEL AS A FUNCTION OF TEMPERING TEMPERATURE FOR TWO DIFFERENT HYDROGEN CONTENTS. TEST TEMP: R.T.



○ MEDIUM HYDROGEN CONTENT: 447 MA. PER SQ. IN. FOR 1/2 HR.

● HIGH HYDROGEN CONTENT: 447 MA. PER SQ. IN. FOR 2-1/2 HR.

( ) BROKE UNDER HEAD

BATH: 10% NaOH AT 30°C.

FIG. 8 TENSILE STRENGTH OF HYDROGEN EMBRITTLED 0.3 IN. DIA. TENSILE SPECIMENS OF 4340 STEEL AS A FUNCTION OF TEMPERING TEMPERATURE FOR THREE DIFFERENT STRAIN RATES. TEST TEMP: R.T.

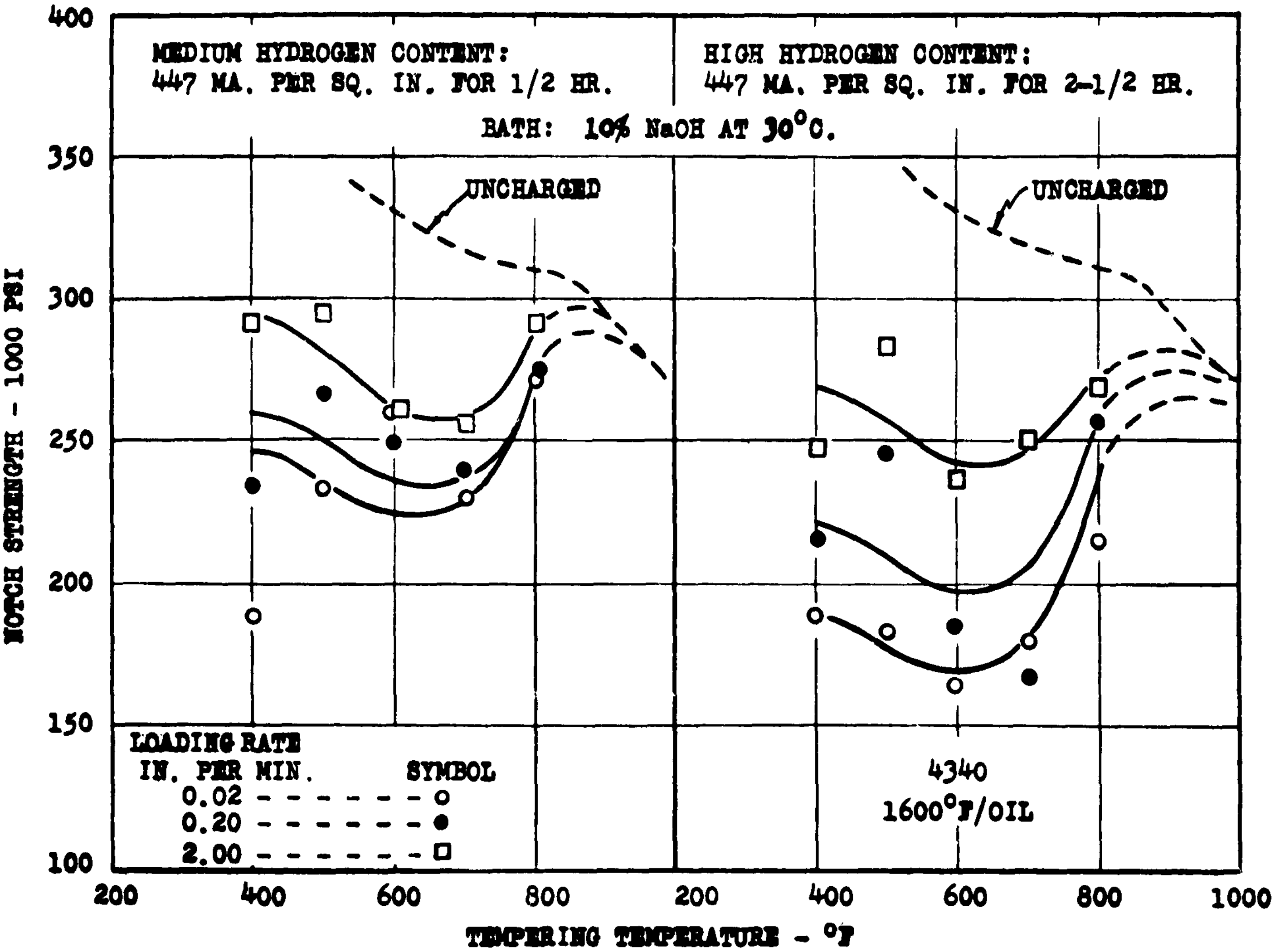


FIG. 9 NOTCH STRENGTH OF HYDROGEN EMBRITTLED 0.3 IN. DIA. NOTCHED TENSILE SPECIMENS (K = 3) OF 4340 STEEL AS A FUNCTION OF TEMPERING TEMPERATURE FOR TWO DIFFERENT HYDROGEN CONTENTS. TEST TEMP: R.T.

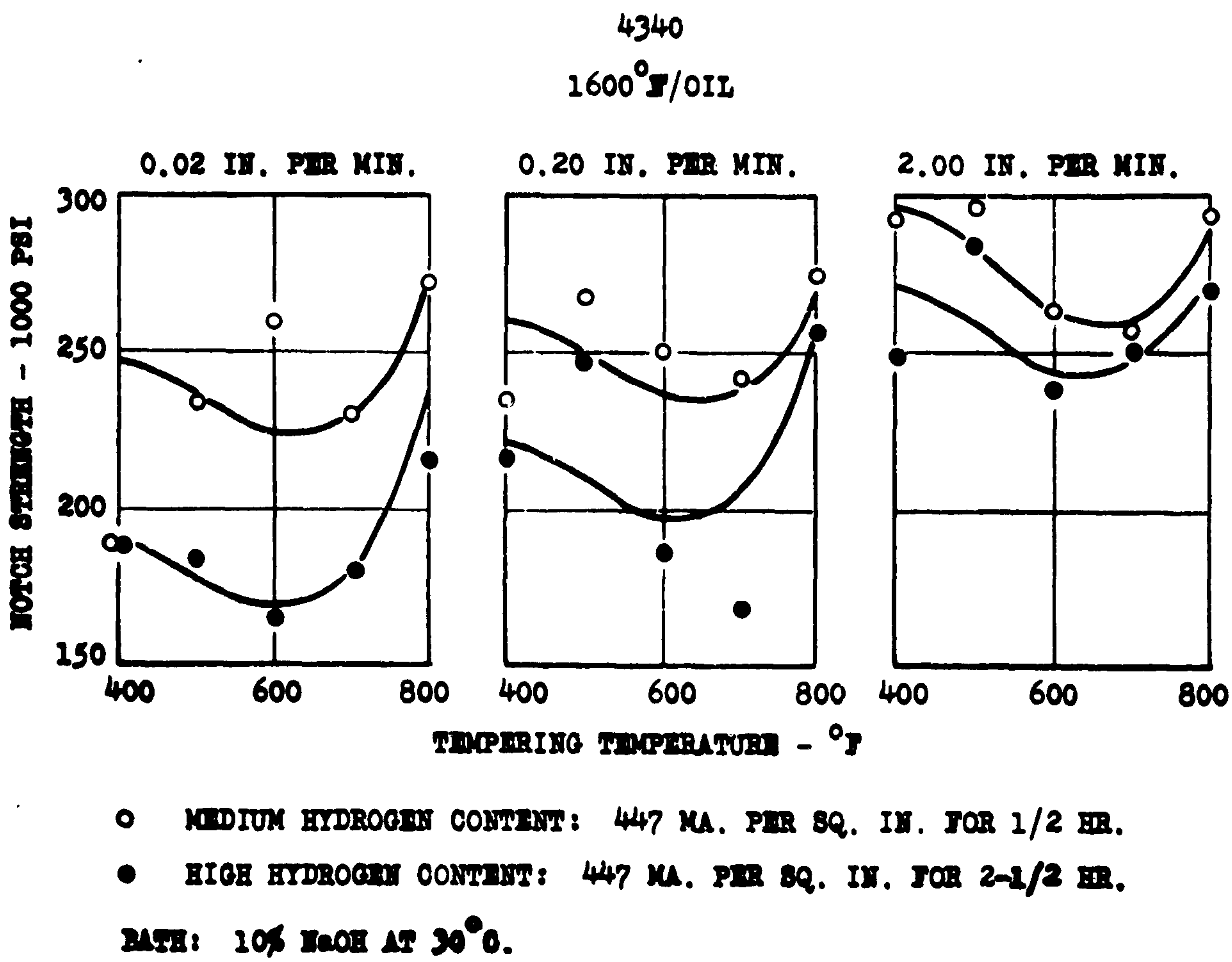


FIG. 10 NOTCH STRENGTH OF HYDROGEN EMBRITTLED 0.3 IN. DIA. NOTCHED TENSILE SPECIMENS (K = 3) OF 4340 STEEL AS A FUNCTION OF TEMPERING TEMPERATURE FOR THREE DIFFERENT STRAIN RATES. TEST TEMP: R.T.

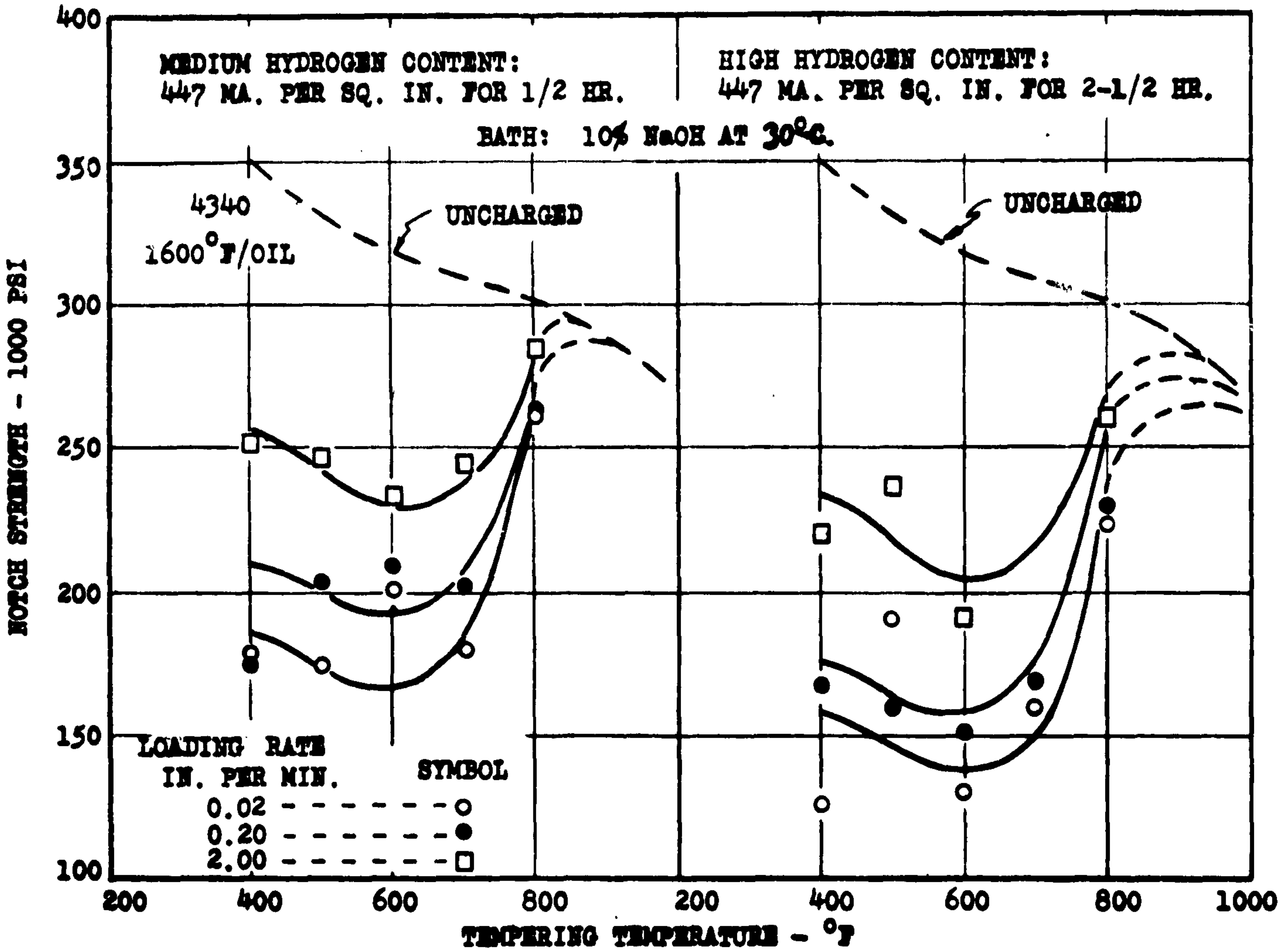
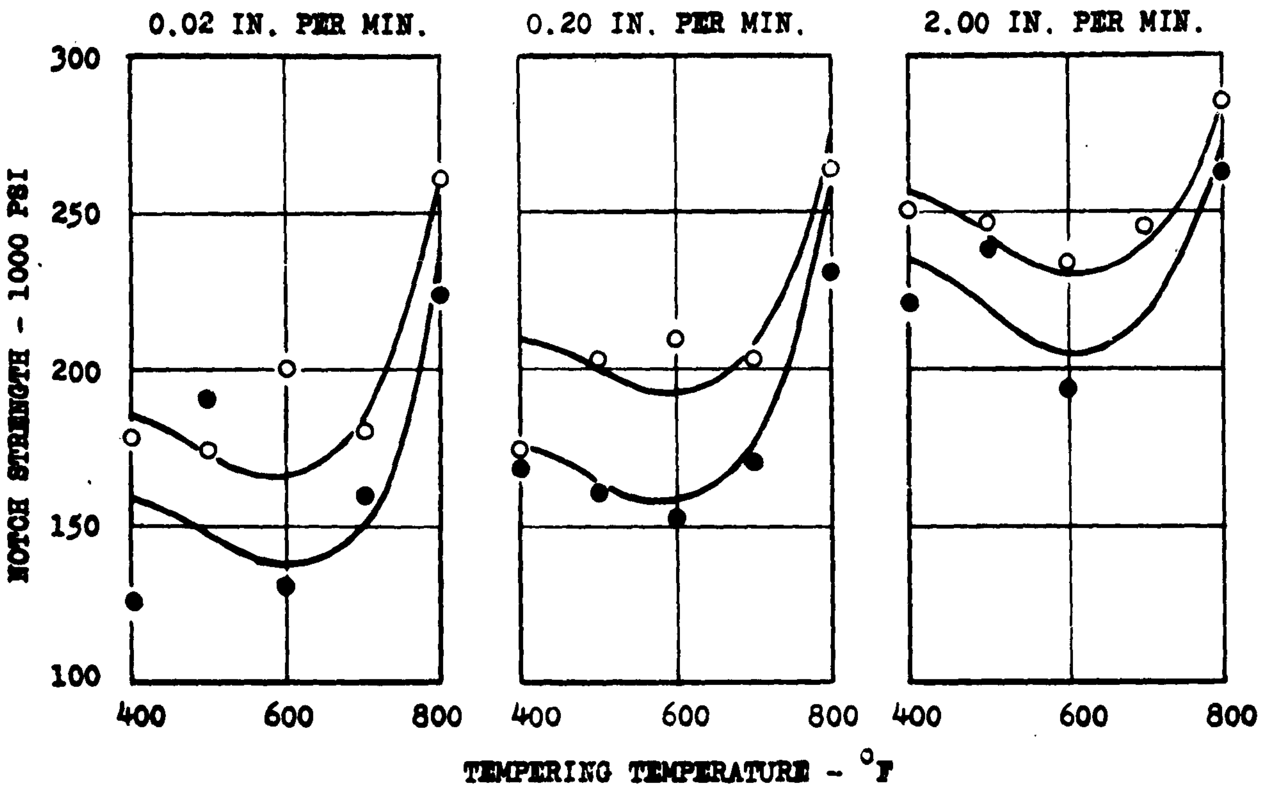


FIG. 11 NOTCH STRENGTH OF HYDROGEN EMBRITTLED 0.3 IN. DIA. NOTCHED TENSILE SPECIMENS (K = 5) OF 4340 STEEL AS A FUNCTION OF TEMPERING TEMPERATURE FOR TWO DIFFERENT HYDROGEN CONTENTS. TEST TEMP: R.T.

4340

1600°F/OIL



○ MEDIUM HYDROGEN CONTENT: 447 MA. PER SQ. IN. FOR 1/2 HR.

● HIGH HYDROGEN CONTENT: 447 MA. PER SQ. IN. FOR 2-1/2 HR.

BATH: 10% NaOH AT 30°C.

FIG. 12 NOTCH STRENGTH OF HYDROGEN EMBRITTLED 0.3 IN. DIA. NOTCHED TENSILE SPECIMENS (K = 5) OF 4340 STEEL AS A FUNCTION OF TEMPERING TEMPERATURE FOR THREE DIFFERENT STRAIN RATES. TEST TEMP: R.T.

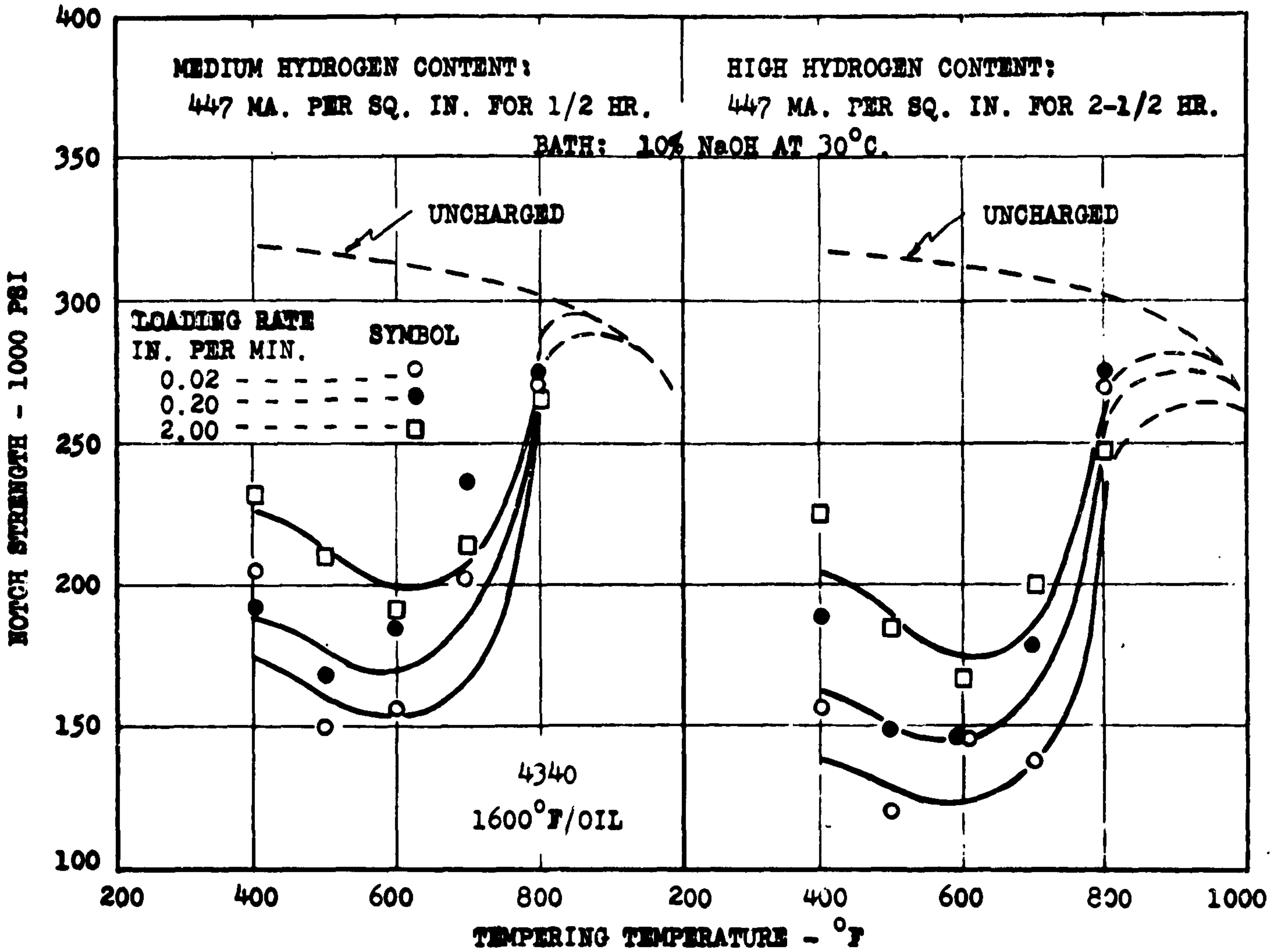
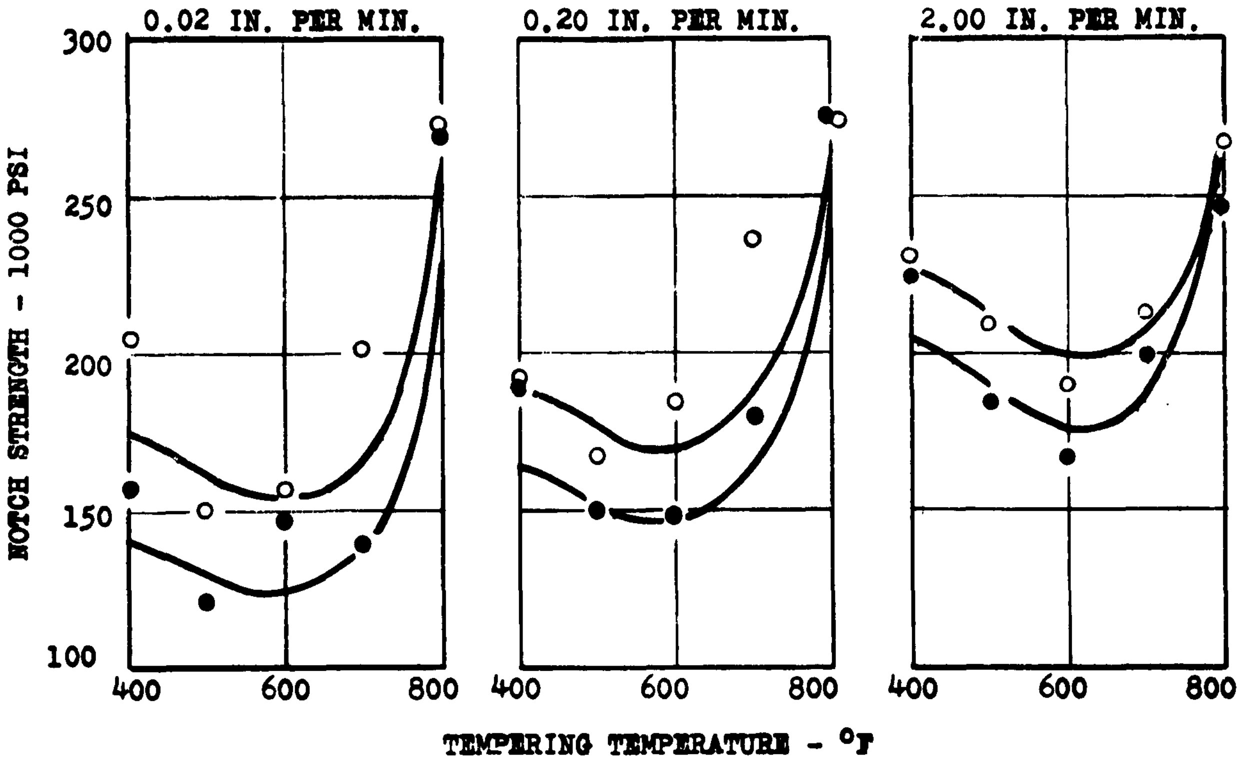


FIG. 13 NOTCH STRENGTH OF HYDROGEN EMBRITTLED 0.3 IN. DIA. NOTCHED TENSILE SPECIMENS (K = 10) OF 4340 STEEL AS A FUNCTION OF TEMPERING TEMPERATURE FOR TWO DIFFERENT HYDROGEN CONTENTS. TEST TEMP: R.T.

4340
1600°F/OIL



○ MEDIUM HYDROGEN CONTENT: 447 MA. PER SQ. IN. FOR ½ HR.

● HIGH HYDROGEN CONTENT: 447 MA. PER SQ. IN. FOR 2½ HR.

BATH: 10% NaOH AT 30°C.

FIG. 14 NOTCH STRENGTH OF HYDROGEN EMBRITTLED 0.3 IN. DIA. NOTCHED TENSILE SPECIMENS (K= 10) OF 4340 STEEL AS A FUNCTION OF TEMPERING TEMPERATURE FOR THREE DIFFERENT STRAIN RATES. TEST TEMP: R.T.

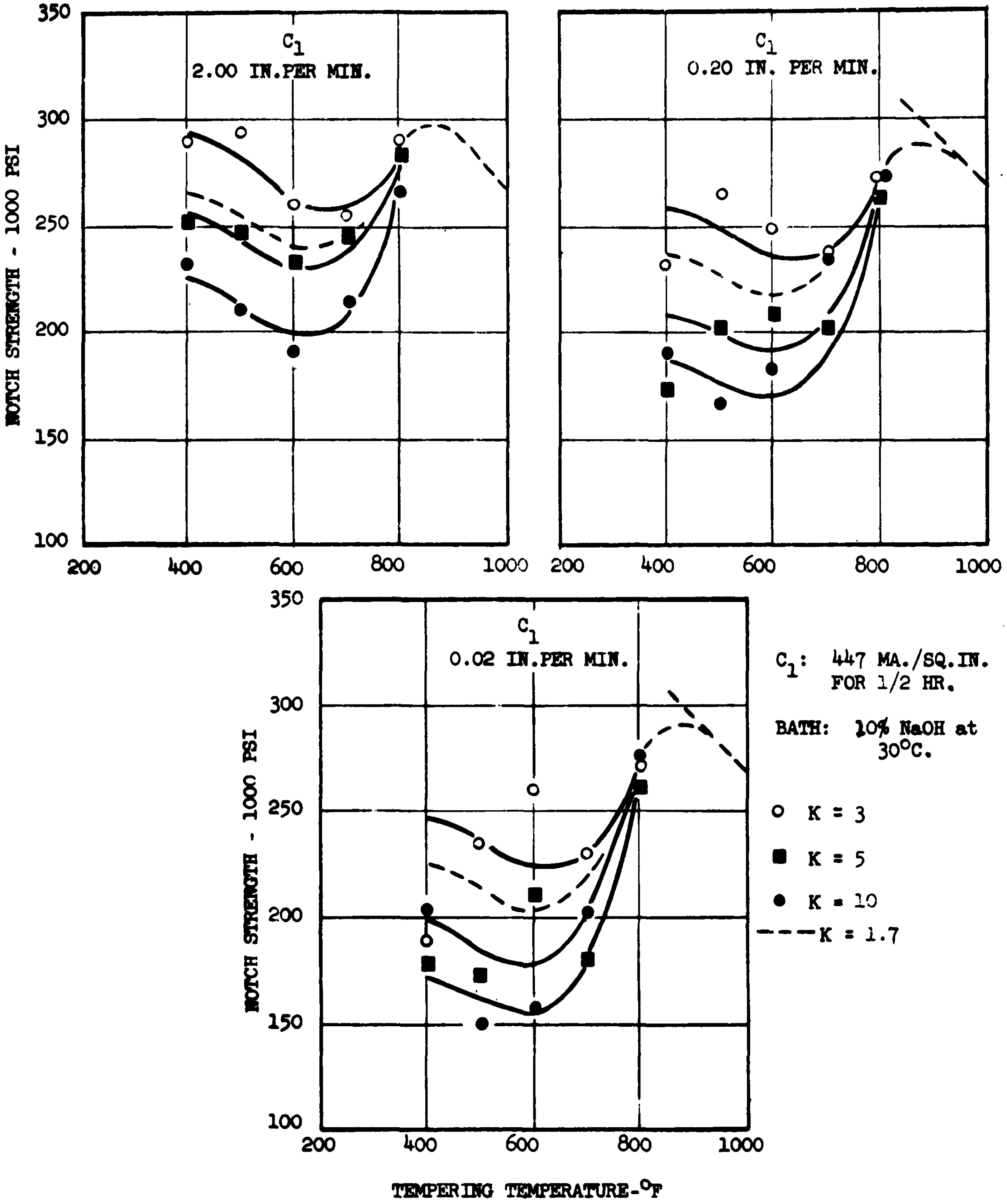


FIG. 15 NOTCH STRENGTH OF HYDROGEN EMBRITTLED 0.3 IN. DIA. NOTCHED TENSILE SPECIMENS OF 4340 STEEL AS A FUNCTION OF TEMPERING TEMPERATURE FOR THREE DIFFERENT STRAIN RATES. TEST TEMP: R.T.

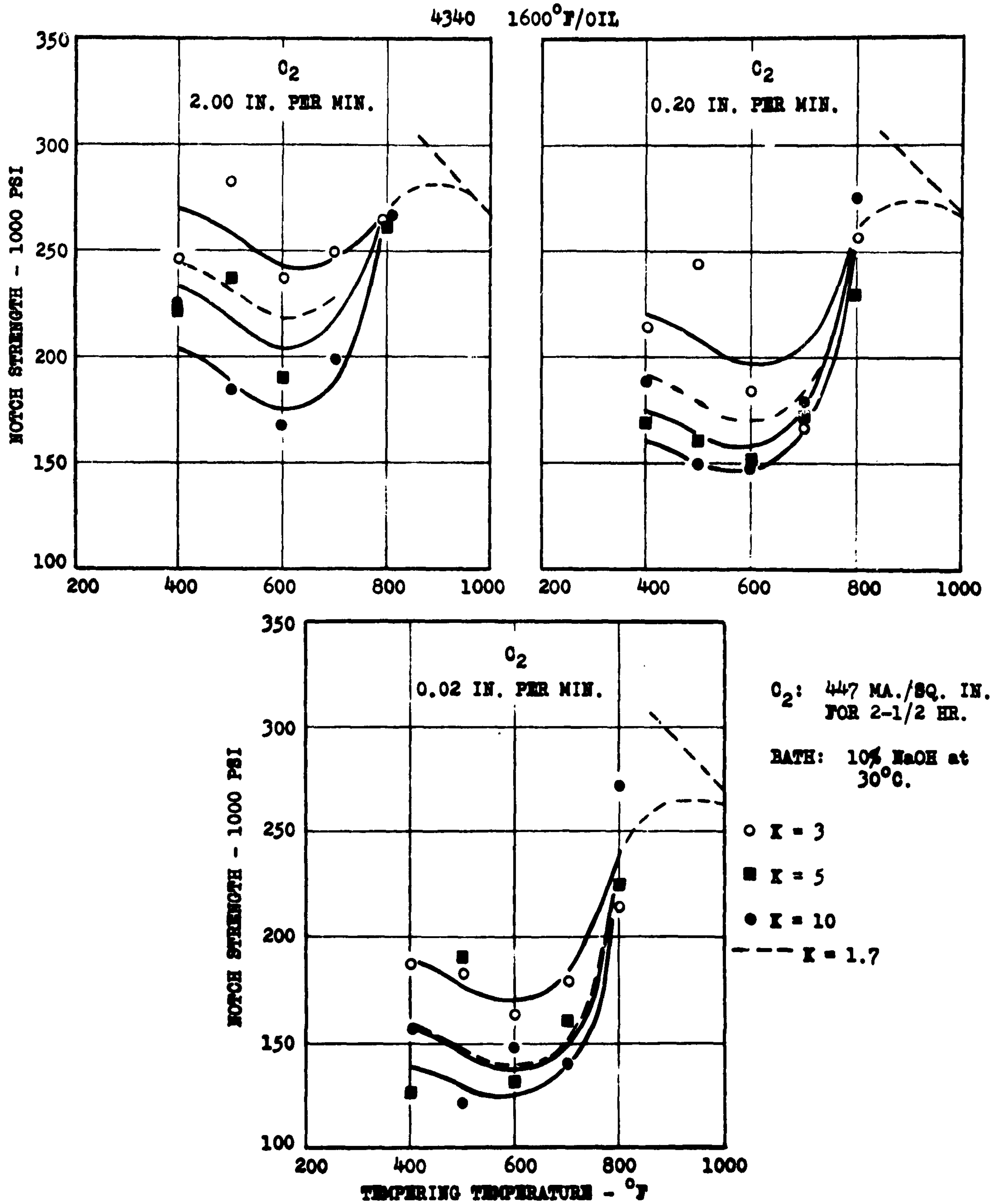


FIG. 16 NOTCH STRENGTH OF HYDROGEN EMBRITTLED 0.3 IN. DIA. NOTCHED TENSILE SPECIMENS OF 4340 STEEL AS A FUNCTION OF TEMPERING TEMPERATURE FOR THREE DIFFERENT STRAIN RATES. TEST TEMP: R.T.

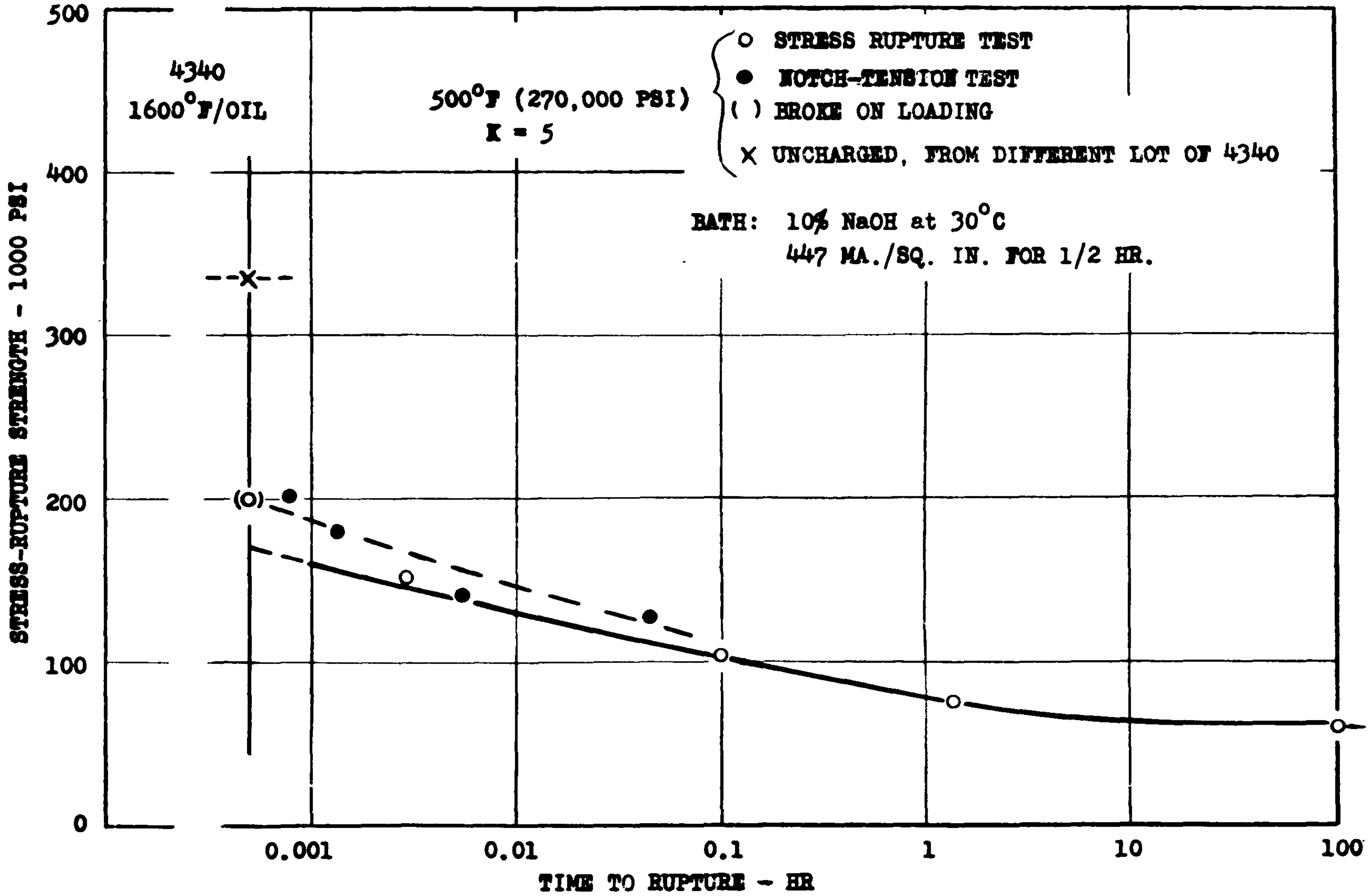


FIG. 17 STRESS RUPTURE DIAGRAM FOR HYDROGEN EMBRITTLED 0.3 IN.-DIA. NOTCHED SPECIMENS (K = 5) OF 4340 STEEL. TEST TEMP: R.T.

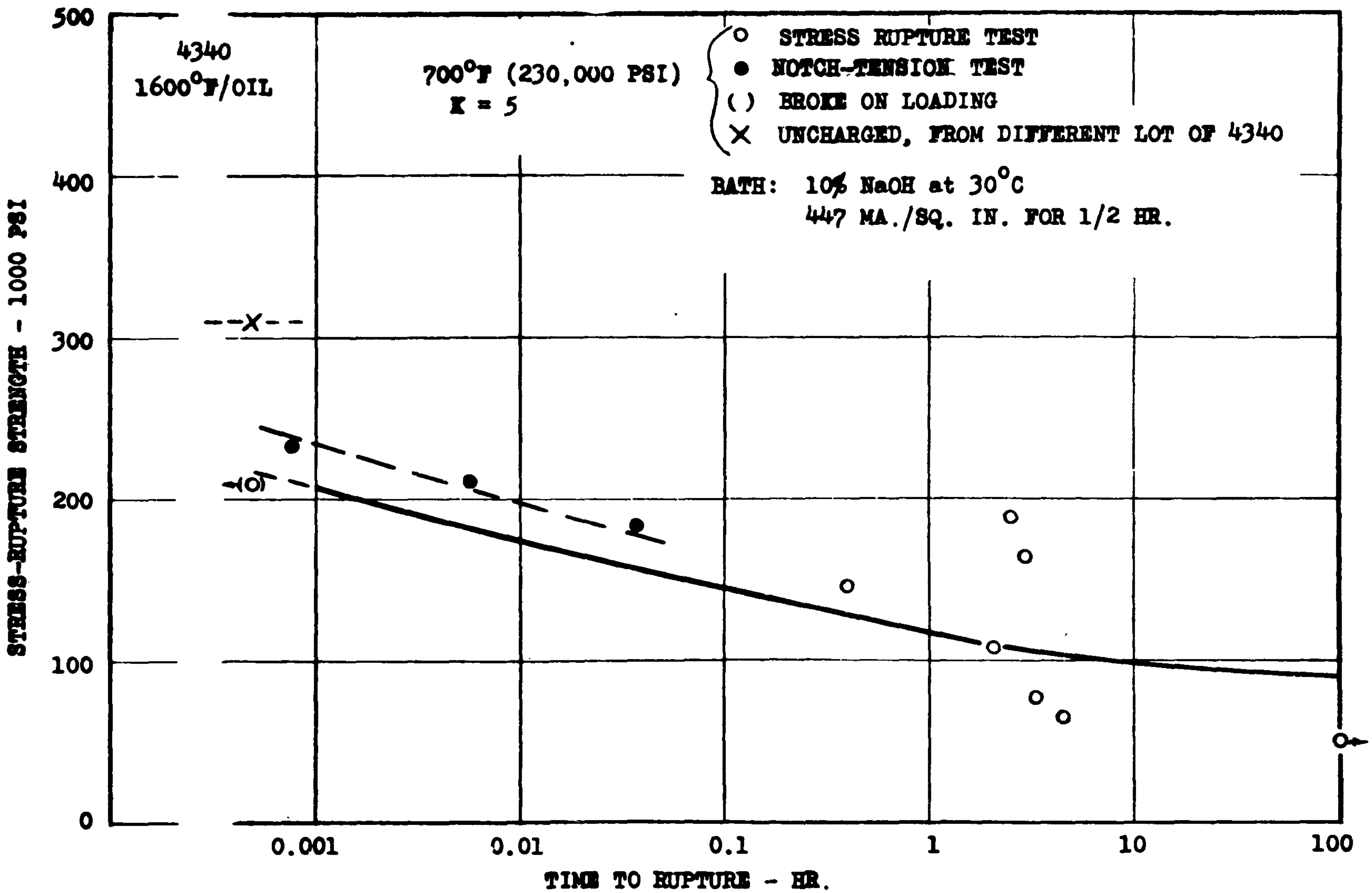


FIG. 18 STRESS RUPTURE DIAGRAM FOR HYDROGEN EMBRITTLED 0.3 IN.-DIA. NOTCHED SPECIMENS (K = 5) OF 4340 STEEL. TEST TEMP: R.T.

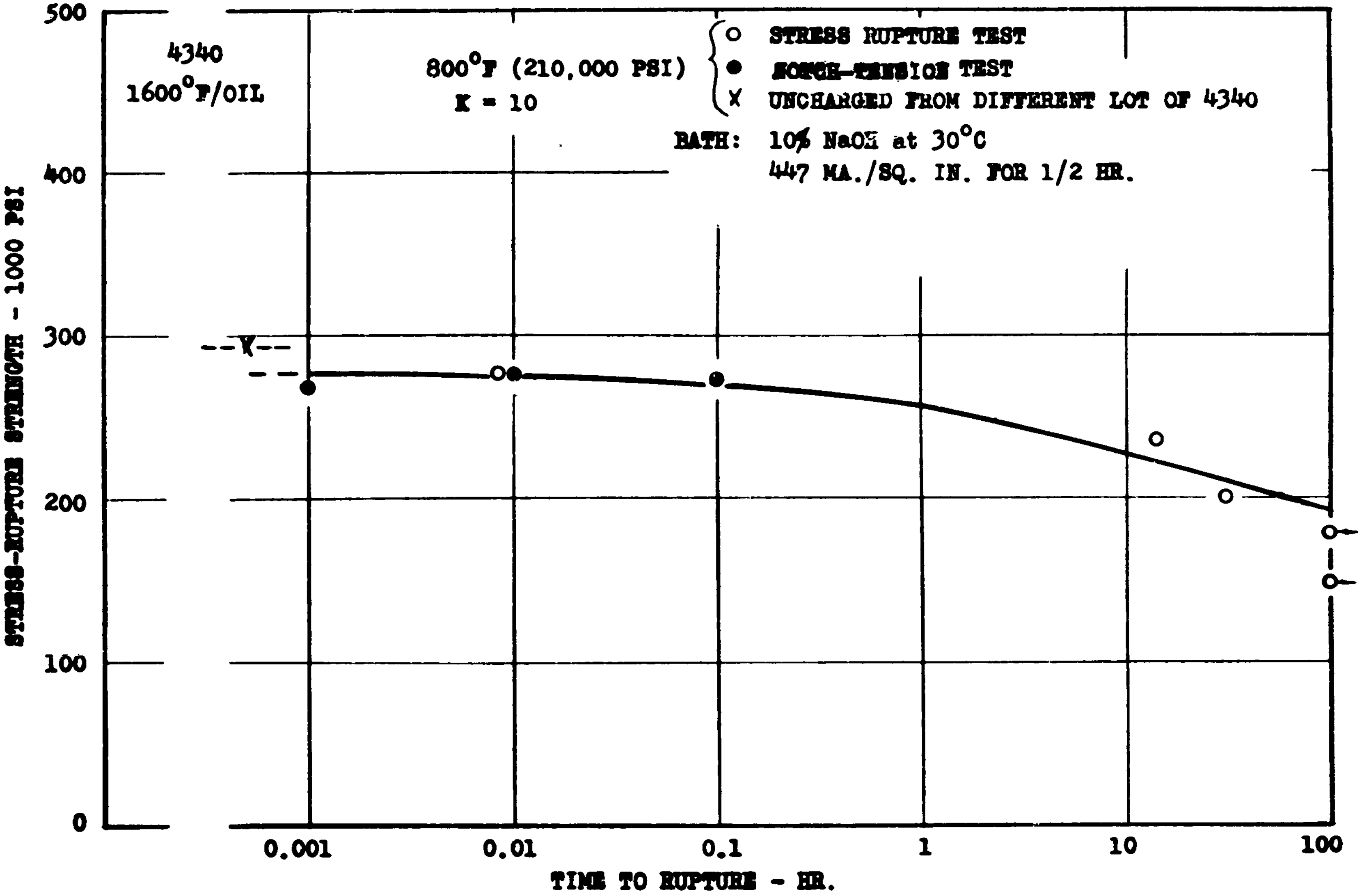


FIG. 19 STRESS RUPTURE DIAGRAM FOR HYDROGEN EMBRITTLED 0.3 IN.-DIA. NOTCHED SPECIMENS (K = 10) OF 4340 STEEL. TEST TEMP: R.T.

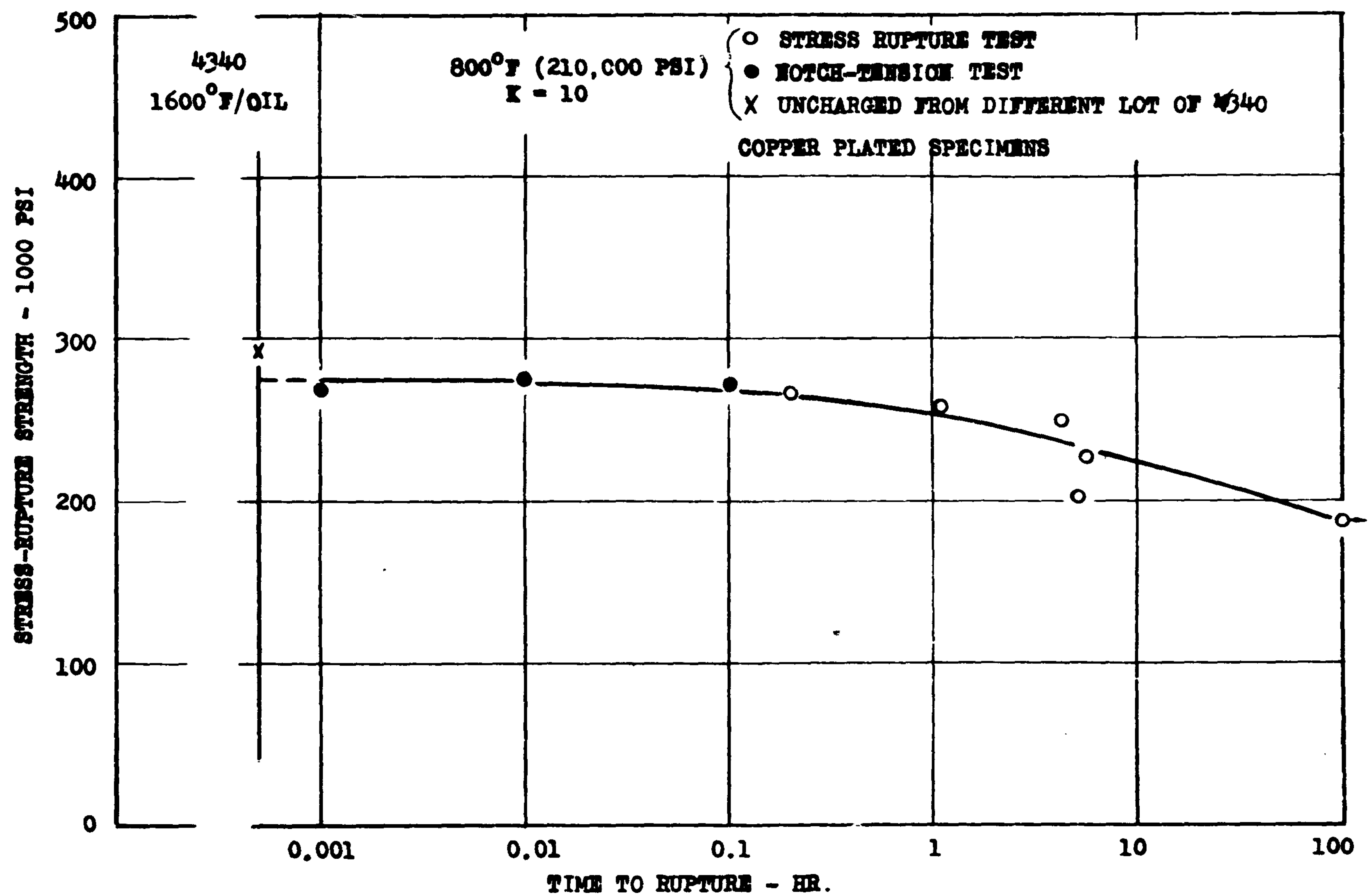


FIG. 20 STRESS RUPTURE DIAGRAM FOR COPPER PLATED 0.3 IN.-DIA. NOTCHED SPECIMENS (K = 10) OF 4340 STEEL. TEST TEMP: R.T.

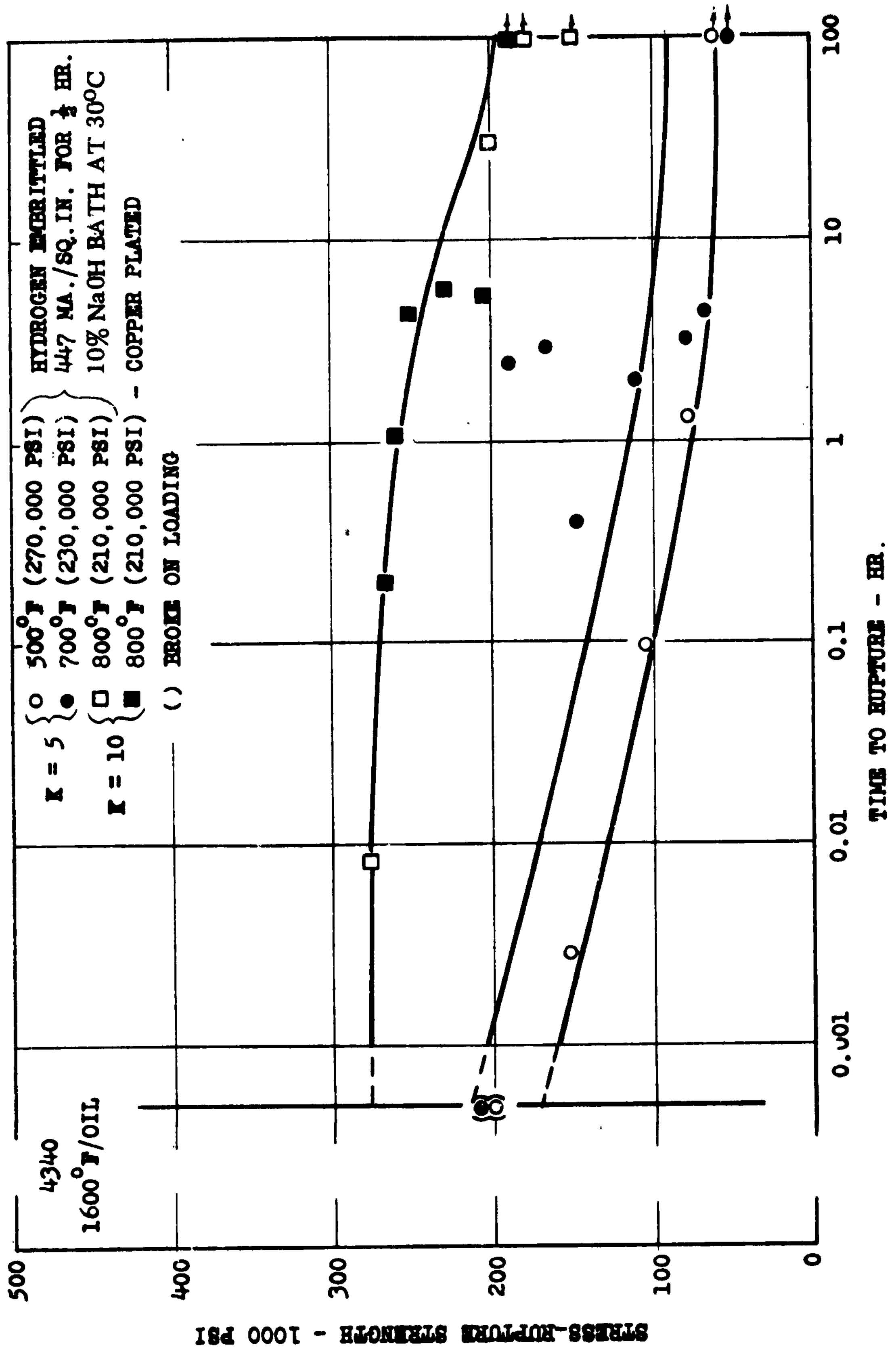


FIG. 21 STRESS RUPTURE DIAGRAM FOR HYDROGEN EMBRITTLED AND COPPER PLATED 0.3 IN.-DIA. NOTCHED SPECIMENS OF 4340 STEEL. TEST TEMP: R.T.

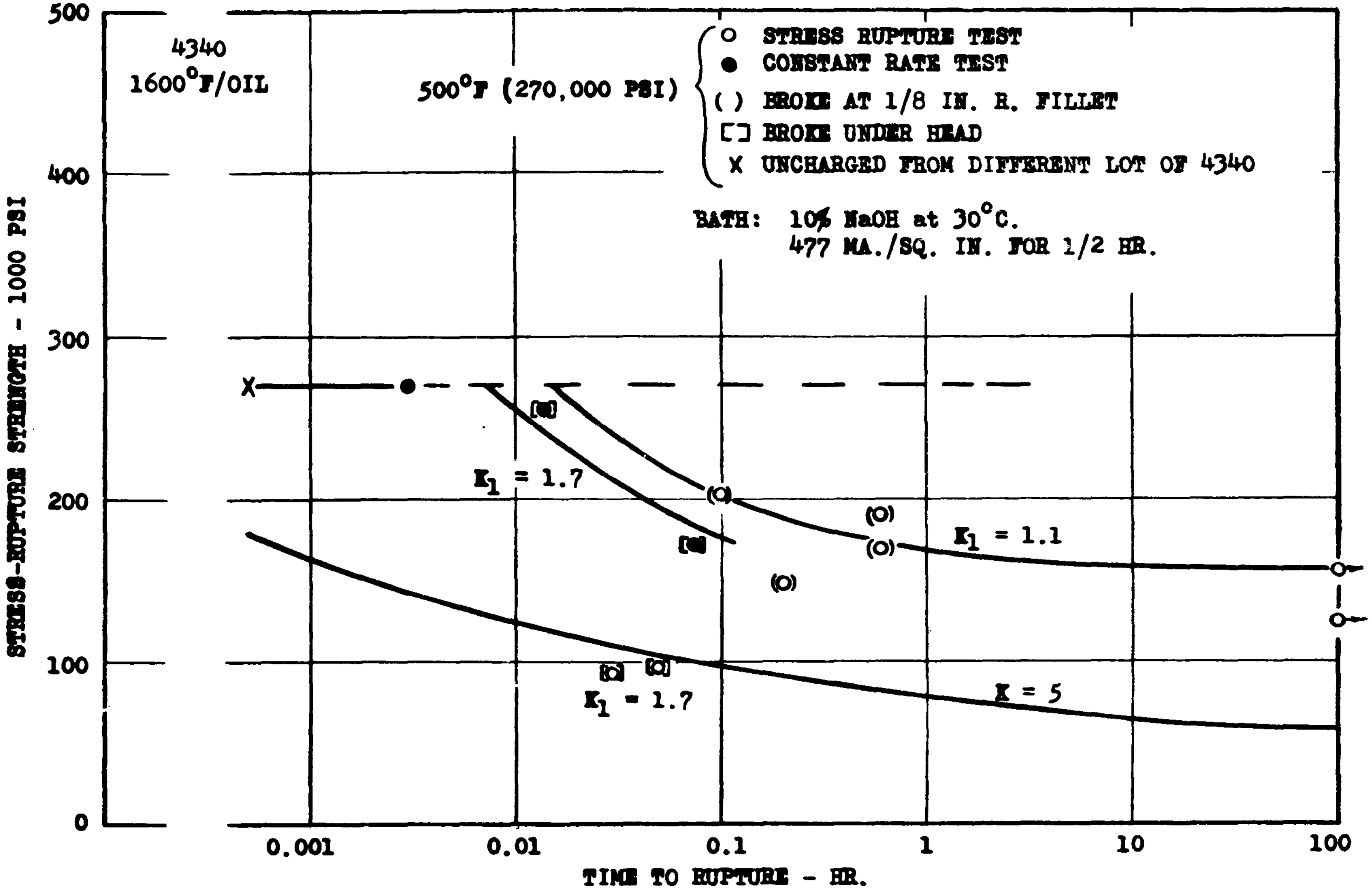


FIG. 22 STRESS RUPTURE DIAGRAM FOR HYDROGEN EMBRITTLED 0.2 IN.-DIA. SMOOTH AND 0.3 IN.-DIA. NOTCHED SPECIMENS OF 4340 STEEL. TEST TEMP: R.T.

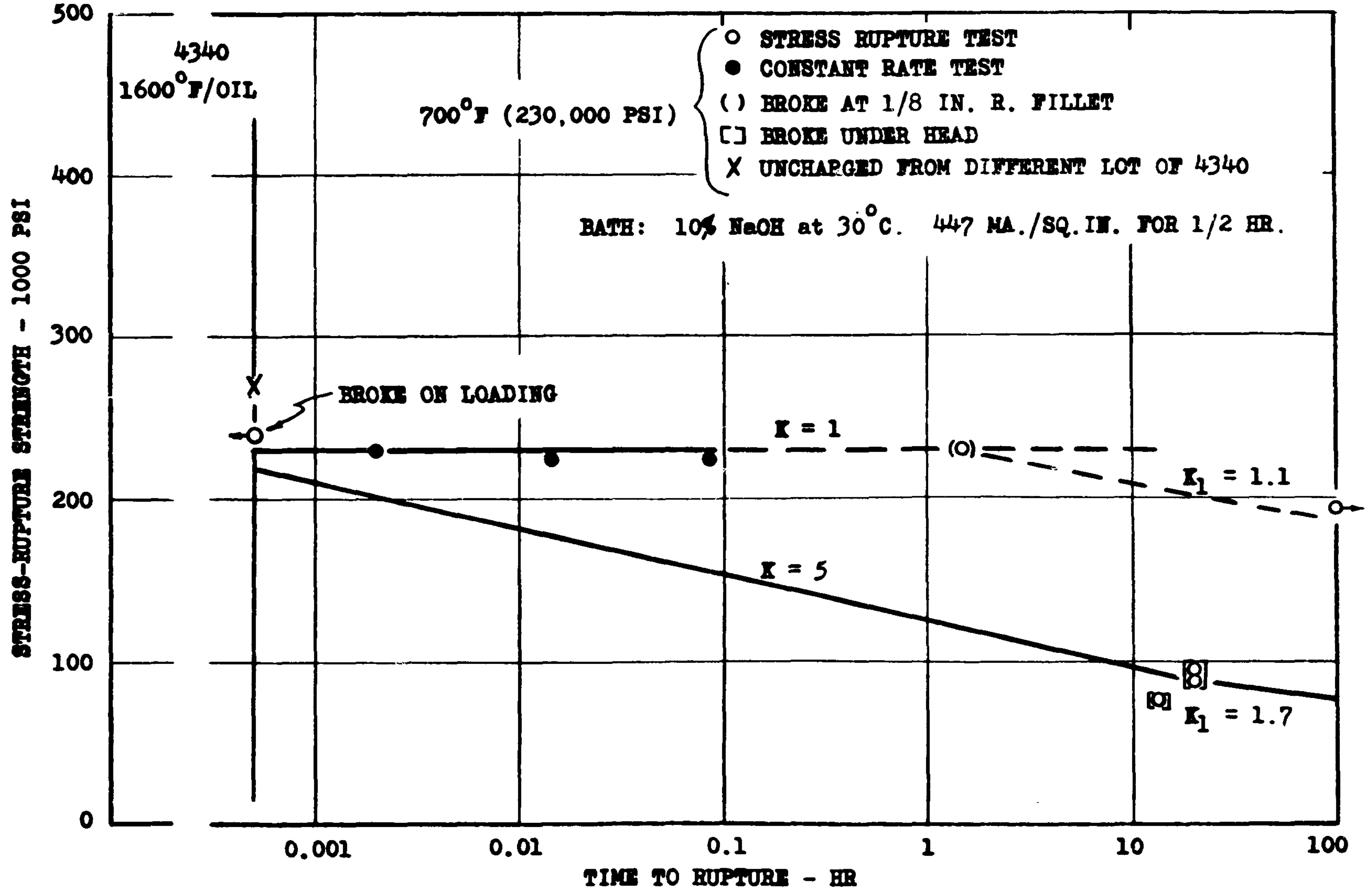


FIG. 23 STRESS RUPTURE DIAGRAM FOR HYDROGEN EMBRITTLED 0.2 IN.-DIA. SMOOTH AND 0.3 IN.-DIA. NOTCHED SPECIMENS OF 4340 STEEL. TEST TEMP: R.T.

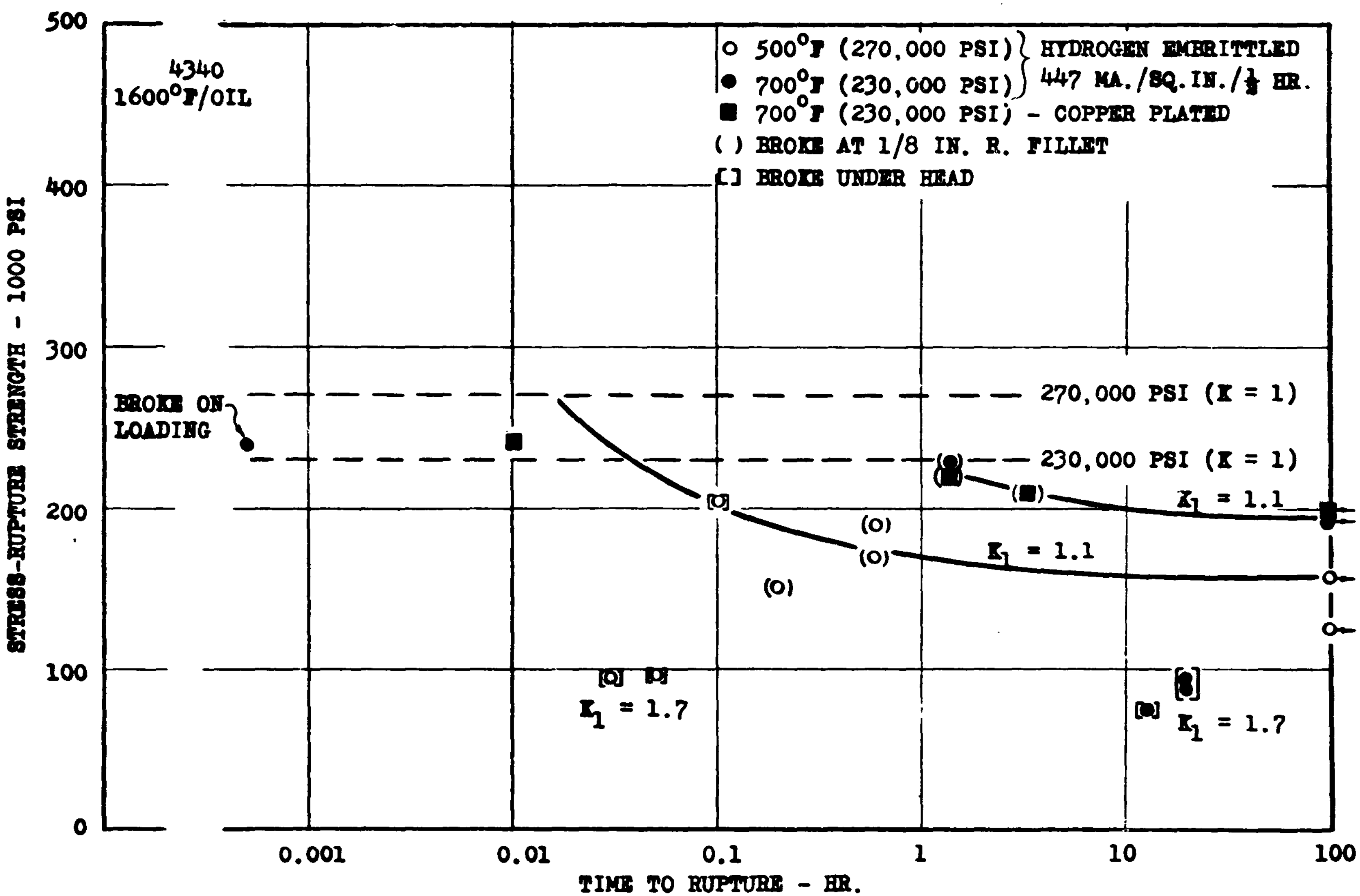


FIG. 24 STRESS RUPTURE DIAGRAM FOR HYDROGEN EMBRITTLED AND COPPER PLATED 0.2 IN.-DIA. SMOOTH SPECIMENS OF 4340 STEEL. TEST TEMP: R.T.

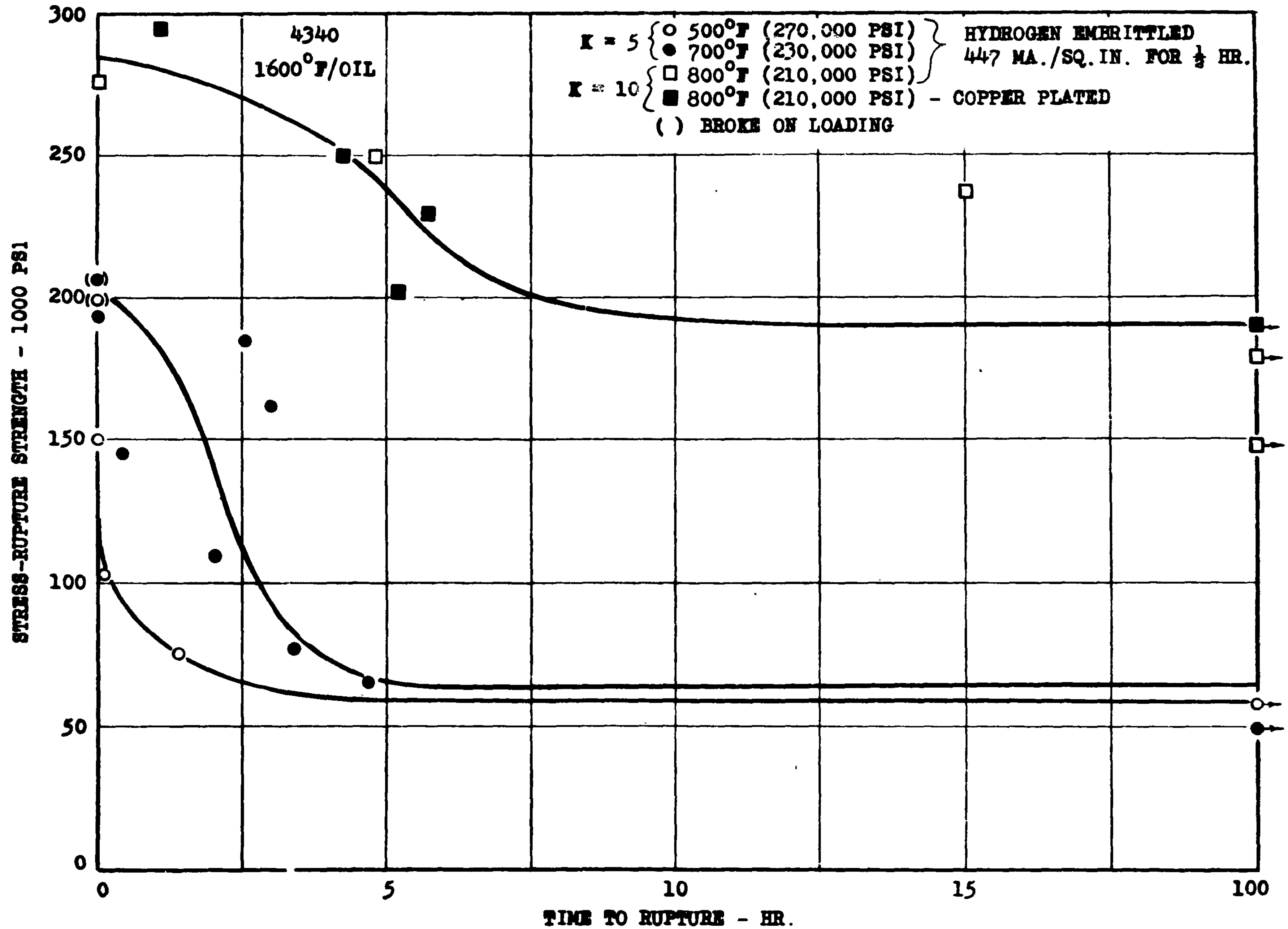


FIG. 25 LINEAR STRESS RUPTURE DIAGRAM FOR HYDROGEN EMBRITTLED AND COPPER PLATED 0.3 IN.-DIA. NOTCHED SPECIMENS OF 4340 STEEL. TEST TEMP: R.T.

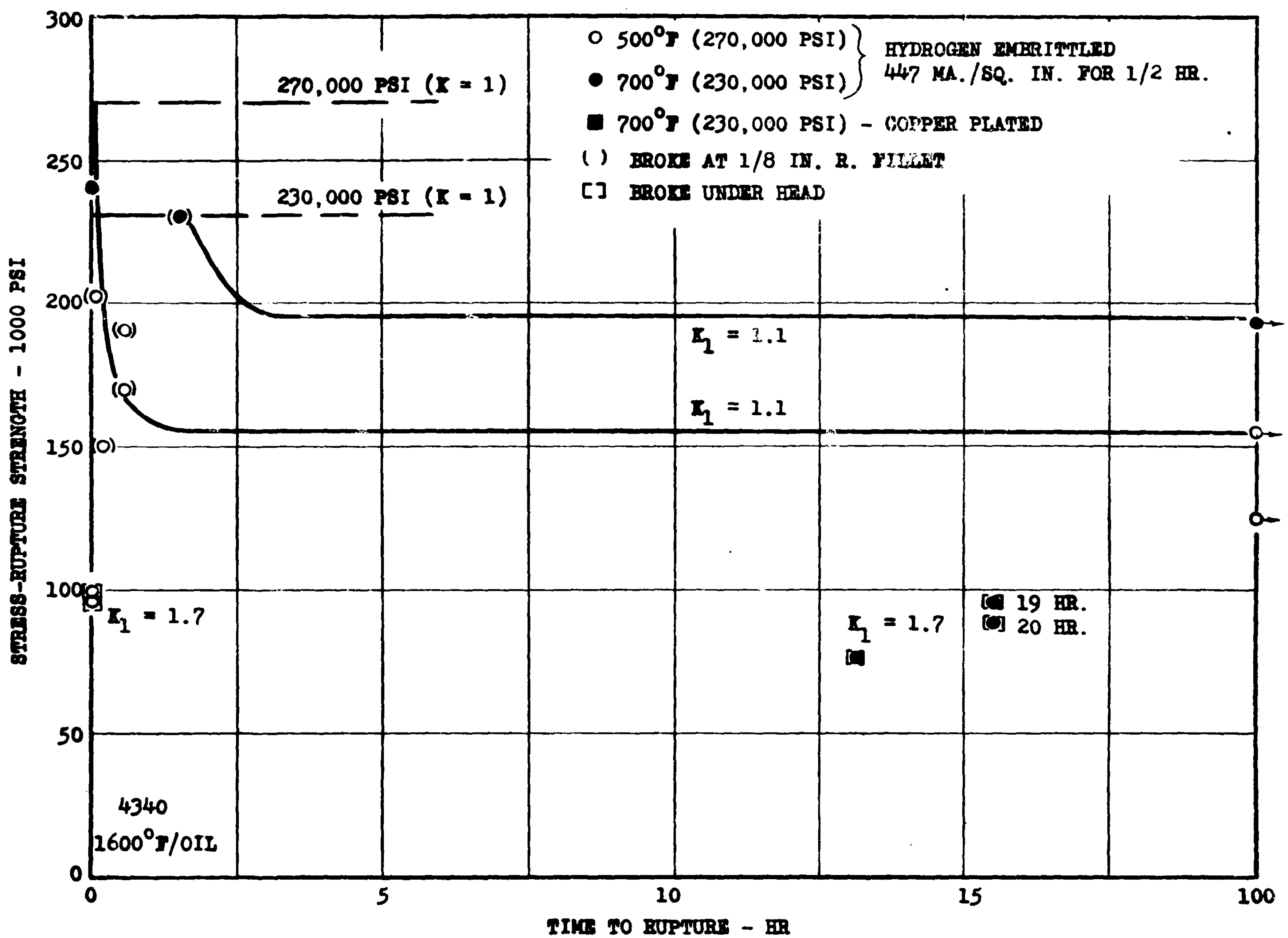


FIG. 26 LINEAR STRESS-RUPTURE DIAGRAM FOR HYDROGEN EMBRITTLED AND COPPER-PLATED 0.2 IN. DIA. SMOOTH SPECIMENS OF 4340 STEEL. TEST TEMP: R.T.

SMOOTH SPECIMEN

NOTCHED SPECIMEN

FIG. 27 FRACTURE ZONES OF EMBRITTLED (a) NOTCHED SPECIMENS AND (b) SMOOTH SPECIMENS UNDER SUSTAINED-LOAD CONDITIONS.

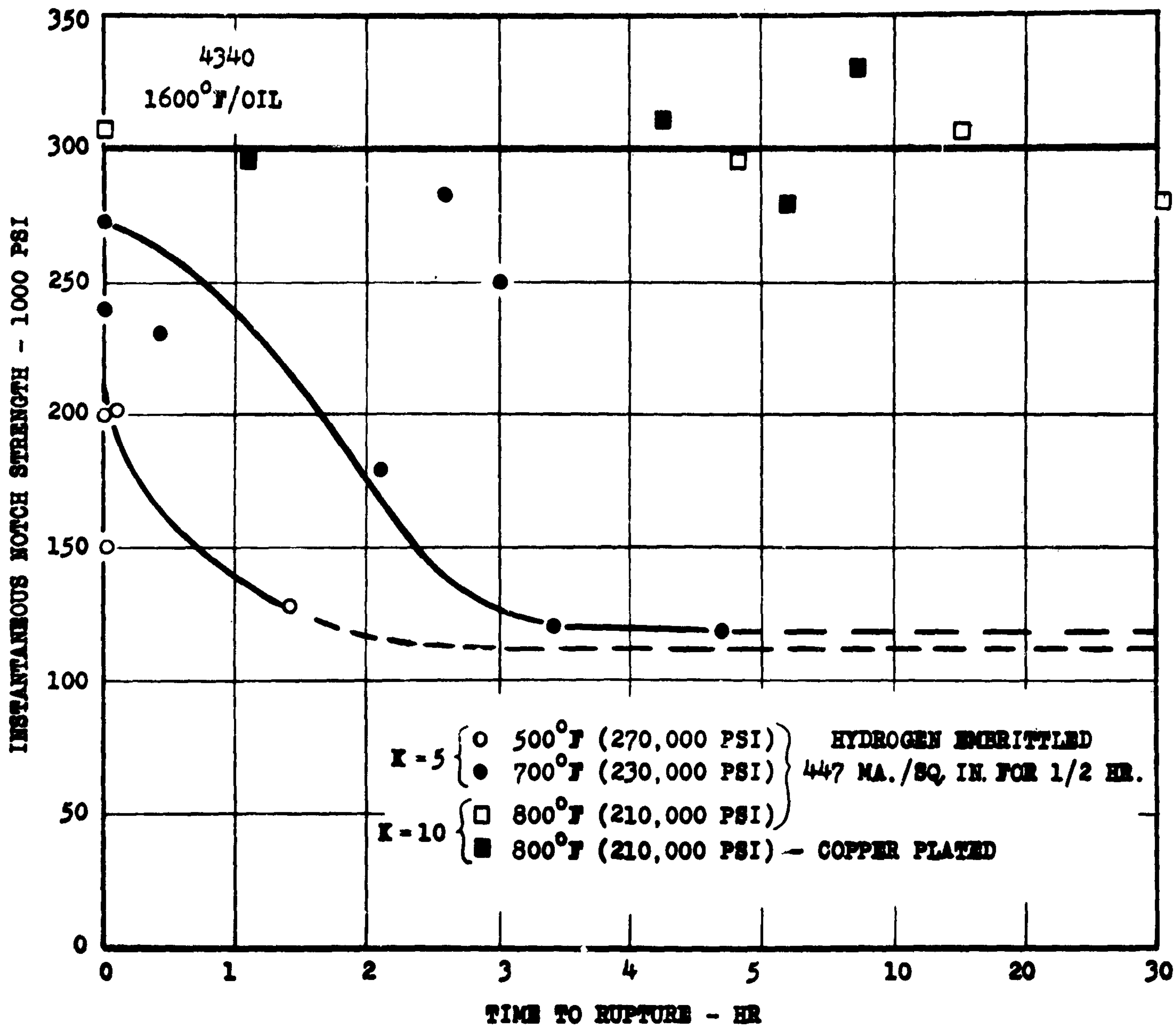


FIG. 28 NOTCH STRENGTH REFERRED TO INSTANTANEOUS FRACTURE AREA AS A FUNCTION OF RUPTURE TIME FOR HYDROGEN EMBRITTLED AND COPPER-PLATED, 0.3 IN.-DIA.-NOTCHED SPECIMENS OF 4340 STEEL. TEST TEMP: R.T.

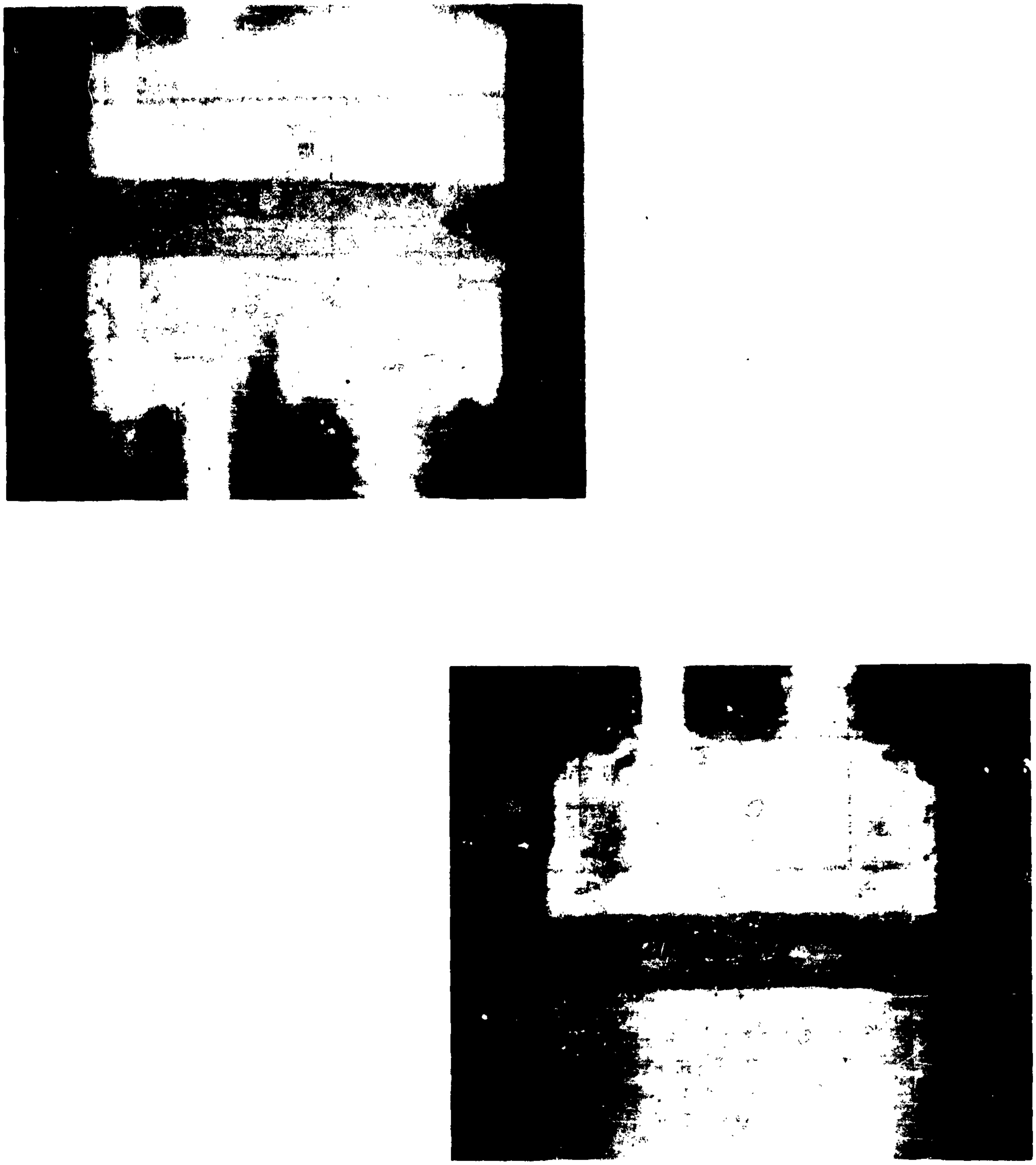


FIG. 29 CRACK DEVELOPMENT IN NOTCHED COPPER-PLATED SPECIMENS UNDER TWO SUSTAINED-LOAD CONDITIONS.

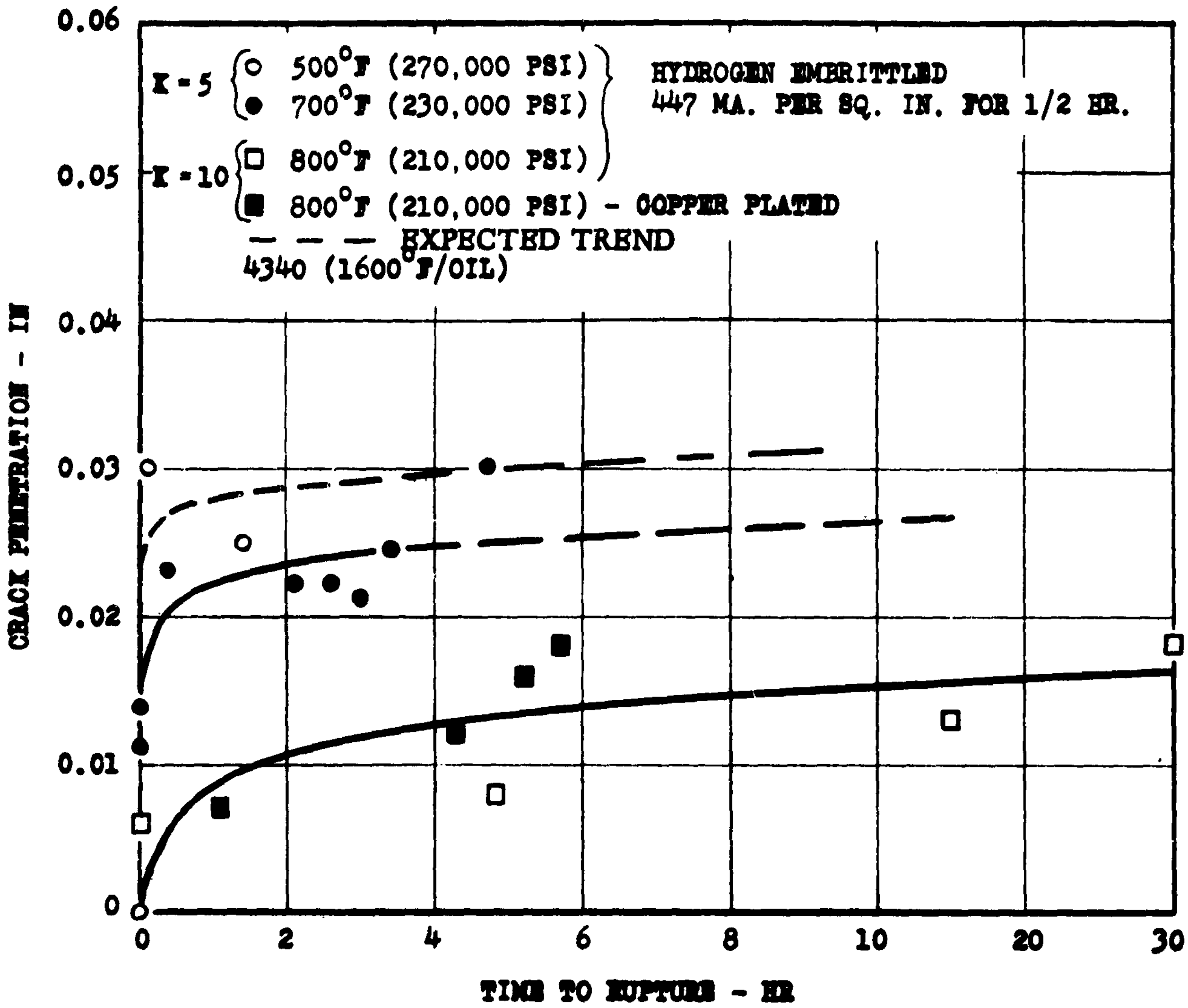


FIG. 30 CRACK PENETRATION AS A FUNCTION OF TIME TO RUPTURE FOR HYDROGEN-EMBRITTLED AND COPPER-PLATED, 0.3-IN.DIA., NOTCHED SPECIMENS OF 4340 STEEL. TEST TEMP: R.T.

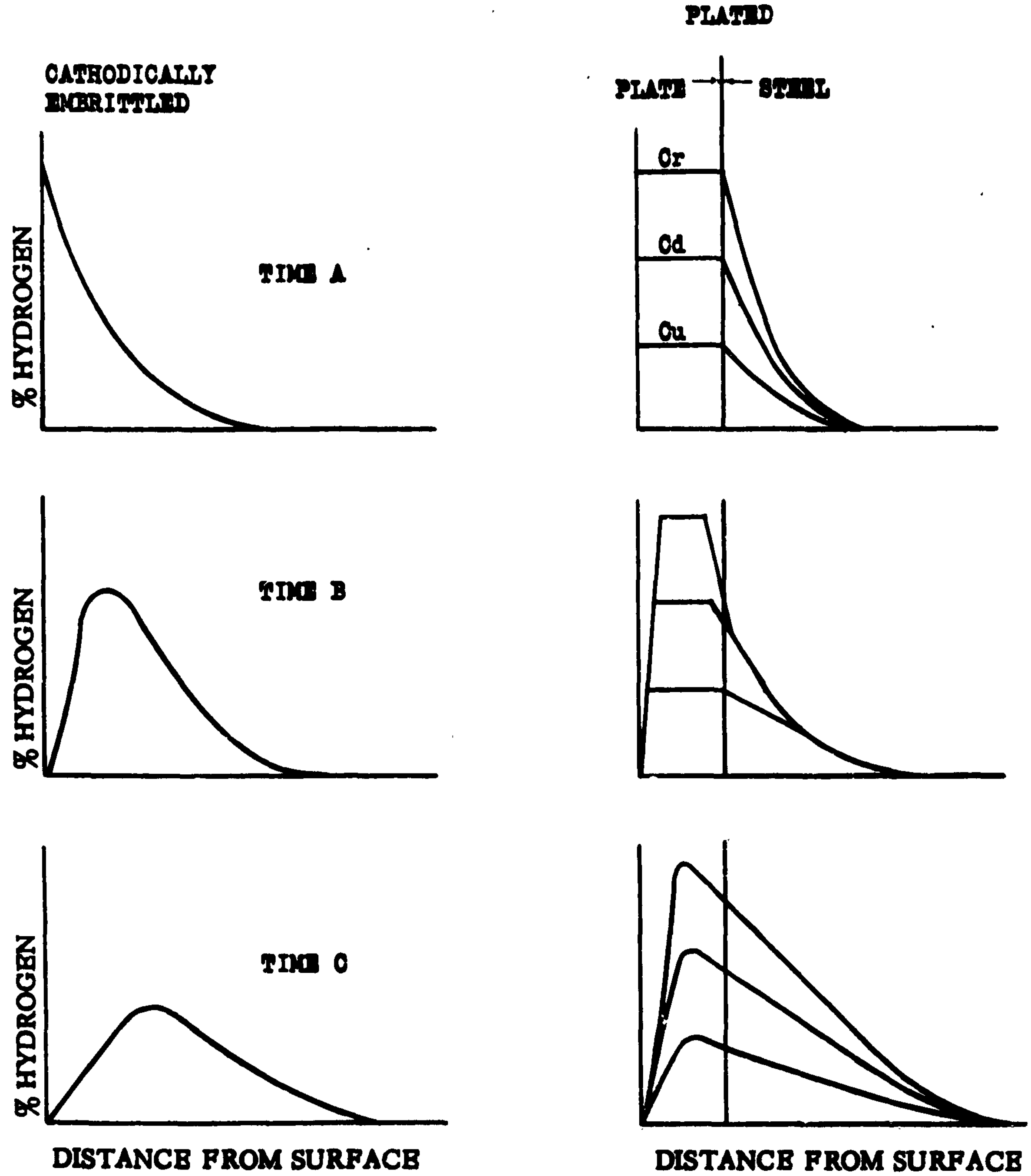


FIG. 31 EXPECTED HYDROGEN CONCENTRATION GRADIENTS IN CATHODICALLY EMBRITTLED AND PLATED SPECIMENS AFTER REMOVAL FROM THE ELECTROLYTIC CELL.

TIME A $<$ TIME B $<$ TIME C.